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GOVERNMENT OF INDIA ATOMIC ENERGY COMMISSION

BARC STUDIES IN COLD FUSION

(April - September 1989)

Edited by P.K. Iyengar and M. Srinivasan

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Cold Fusion Experiments at Hall No.2: Experimental Results of Heavy Water Electrolysis using a Commercial Pd—Ni Electrolyser

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INTRODUCTION:

When the news about the occurrence of cold fusion broke—out1,2, it was decided to conduct similar experiments at Heavy Water Division, BARC in collaboration with Neutron Physics Division. Due to the urgency of the work it was felt necessary to employ a readily available electrolyzer with minimum modification so as not to waste minimum time in setting up of the experiments. Fortunately, a commercial laboratory hydrogen generator was available in a ready to use condition. It was therefore decided to conduct the initial experiments with this electrolyzer. Both neutron and tritium measurements ere carried out. This paper gives a detailed account of these experiments and the results obtained are discussed.

The Electrolyzer:

The electrolyzer employed was a commercially available diffusion type ultrapure electrolytic hydrogen generator. A schematic sketch of the electrolytic cell is shown in Fig.1a. The anode is of Nickel and the cathode consists of specially activated palladium membrane tubes. These anodes and cathodes are mounted as shown in Fig.1b. The outer nickel body of the cell and a central nickel tube serve as coaxial anodes and sixteen cathode tubes are mounted with the help of PTFE spacers in between these two anodes as shown in Fig.1b. All the cathode tubes are sealed at the top and are opening into a small reservoir through which the deuterium comes out. (Fig.1a). Some typical cell parameters are listed in Table (1). During electrolysis the oxygen generated at the anode is vented through a series of plates (for draining any alkali carryover) through a vent tube at the top. The hydrogen ions which deposit at the Pd cathodes, under the influence of applied electric potential diffuse through the walls of the tubes. These ions recombine inside the tubes to form molecule deuterium. The electrolyte level in the electrolytic cell is maintained via remote reservoir of water. The unit is completely automatic and is equipped with pressure control, a solenoid valve, electrolyte leak detector, low water level detector, temperature control etc.

Detectors :

Initially a plastic scintillation detector with PM tube () and an assembly of four BF₃ detectors were used for radiation monitoring. Subsequently He3 detectors were also employed.

Experimental:

To start with, the electrolyzer was operated with 20% NaOH in natural water as the electrolyte as a blank run. A BF₃ counter (efficiency ~10⁻⁴) and plastic scintillator counter (efficiency ~10⁻²) were kept about 10 cm from the cell. This operation was carried out for about 48 hr for collecting the background data. The cell was then drained of its

electrolyte flushed with heavy water and then filled with 20% NaOD solution in D2O (>99.8% isotopic purity) and was operated initially at 30 amps. The current was slowly raised to 60 amps corresponding to a current density of 200 mA/cm2. After an operation under these conditions for about 3 hrs both the neutron counters started showing occasional rise in the neutron counts and after about 5 hrs of operation both the counters suddenly showed two very large peaks and at the time of the second peak the current is the electrolyzer suddenly increased to ~120 amps on its own and the electrolyzer immediately got tripped. Later it was found that the plastic insulation of the electric wire between the DC power supply and the electrolyzer had melted and even the soldering at the connection had melted. The diodes of the power supply had burnt and that is why the unit had tripped. The results of the neutron measurements of this run are shown in Fig.2. The total number of neutrons produced is 108 over a period of several hours of electrolysis, however actual neutron emission was in bursts of 5 minutes and 9 such bursts were observed. During this operation no tritium analysis was carried out but at the end of the run the electrolyte was analyzed for its tritium content by liquid scintillation counting and was found to contain 1.5 µci/ml tritium activity. The tritium activity of the heavy water used for preparing the electrolyte was 0.075 nci/ml. As discussed later this tritium build up is much more than what can be accounted for by the electrolytic enrichment.

The cell was drained of its electrolyte and flushed with heavy water many times for decontaminate from tritium. Finally the electrolyte solution was prepared using heavy water containing 0.075 nci/ml tritium and the electrolyzer charged with this solution and left overnight. Next day this electrolyte was analyzed for its tritium content and now electrolysis was started using an external power supply, as built in power supply of the electrolyzer had got damaged.

The electrolyzer was operated at currents of 60-62 amps (current density ~200 mA cm-2) continuously for 54.5 hrs. Samples of electrolyte were with drawn at different time intervals during this operation for tritium analysis. During this run no significant neutron

peak was observed in any of the detectors.

The tritium content of the electrolyte also did not increase, instead it decreased from 0.32 nci/ml (at 0 hrs) to 0.12 nci/ml at the end of the run. This decrease can be explained easily. The starting sample showed higher tritium content than the D2O employed because of the residual tritium in the electrolyzer from the previous run. Now as the electrolysis proceeded D2O of low activity (0.075 nci/ml) was used for continuously maintaining the level of the electrolyte which was therefore diluting the tritium in the electrolyte. At the end of this run, through the current had been put off, the neutron detectors were still kept on looking at the cell. Nothing happened for the next 30 hrs. Even the tritium content of the sample withdrawn about 24 hrs after putting off the current showed same reading. But about 30 hrs after putting off the current a large neutron burst was observed. Corresponding to about 106 neutrons. As this neutron peak occurred during night, next morning another sample of electrolyte was withdrawn and analyzed for its tritium content. It was found to contain 121 nci/ml tritium. The experiment was stopped at this point but the electrolyser was left as such with the electrolyte in the cell and deuterium at 1 kg/cm2 in its deuterium chamber. After a lapse of about a month the electrolyte was removed and analyzed for tritium. A four fold increase in the tritium was observed. All these results of tritium and neutron measurements are depicted in Fig.3.

The electrolyzer has since been cleaned and is being charged with fresh electrolyte. Another operation of longer duration with online tritium counting both in deuterium gas and the electrolyte line is being arranged to establish a point to point correspondence between the neutron and tritium counting.

Discussion:

The results of the neutron and tritium measurements are tabulated in Table 2. The observed tritium concentrations have been corrected for the tritium enriched due to electrolytic separation of deuterium and tritium and evaporation. In the last two columns of this table the probability of cold fusion occurring via neutron and tritium channel have been listed for both the cases. These have been calculated in terms of the no. of particles (neutron or Tritium) being produced per pair of deuterium atoms absorbed in the metal. A Pd: D ratio of 0.6 has been taken as widely reported in the literature. It is quite important to note from this table that the tritium channel seems to be much more probable than the neutron channel through in 'Hot Fusion' their probability is equal. One point which needs further experimental confirmation is that both the channels seen to be occurring simultaneously because as mentioned earlier in the second run no neutron and tritium peak was observed for a long duration but the tritium content of the electrolyte was found to have increased multifolds after a neutron burst was noticed. As already mentioned this needs further confirmation which can be done by on line measurement of tritium which is being attempted by us at present. Another interesting observation is that electrolysis seems to be necessary only for loading the metal with deuterium. A flowing current does not seen to be a necessary condition for fusion to occur as is clear from the observation in RUN2 of the electrolyzer where both neutron peaks and tritium were recorded only about 30 hrs after the current had been put off. A four fold increase in tritium over a period of is one month further strengthens this view point. Observation of tritium in Pd loaded directly from gas phase also substantiated the conclusion that ongoing electrolysis is not really essential on cold fusion to occur. Another observation from this work is that the Pd metal seems to lose its capability to produce cold fusion with time. As can be seen from a comparison of RUN1 and RUN2 in the latter the no. of neutrons and tritium produced seen to have decreased. This observation is very important and calls for further investigation in terms of some specific parameter(s) of the metal which is important for cold fusion. A multi dimensional characterization including lattice structure etc of fresh and spent electrodes will go a long way in understanding this phenomena and will also probably throw some light on the mechanism of 'Cold Fusion'.

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The authors have been getting unstinted cooperation from several scientists in various aspects of these experiments. They are extremely thankful to each and every one of them. They include SS C.K. Pushpangathan, V.H. Patil, Arun Kumar and N.P Sethuram of Heavy Water Division and Shri R.K. Rout Kum V. Chitra and Shri L.V. Kulkarni of Neutron Physics Division. The all important tritium measurements were carried out by Dr T.S. Iyengar painstakingly, which is gratefully acknowledged. Shri H.K. Sadhukhan, Head Heavy Water Division has contributed immensely by fruitful discussion and authors are thankful to him.

References:

2) S.E. Jones et al; Nature; 338; 737; 1989

¹⁾ M. Fleischmann, S. Pons; J. Electranal. Chem. and Interfacial Electrchem.; 261; 301-308; 1989

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TABLE 1 Details of Electrolytic Cell

Vol. of Pd cathode = 19.98 cm³

Wt. of Pd = 247.95 gm

Area of cathode = 300 cm²

Current = 60 amps

Current density = ~ 200 mA/cm²

Electrolyte = 20% NaOD in D2O

Total vol. of electrolyte = 250 ml

TABLE 2

Summary of Neutron and Tritium Yields (current density = 200 mA/cm²)

(Duration of	Total no. of Electrolysis	neutron in Bursts	Tritium
Run 1 7	72 hrs.		1.3x1Ø-8
Run 2 16.6.89	54 hrs.		*1.Øx1Ø-9 *3.9x1Ø-9
			1

 $[\]ast$ These were obtained 30 hrs. and 27 days respectively after the current had been switched off.

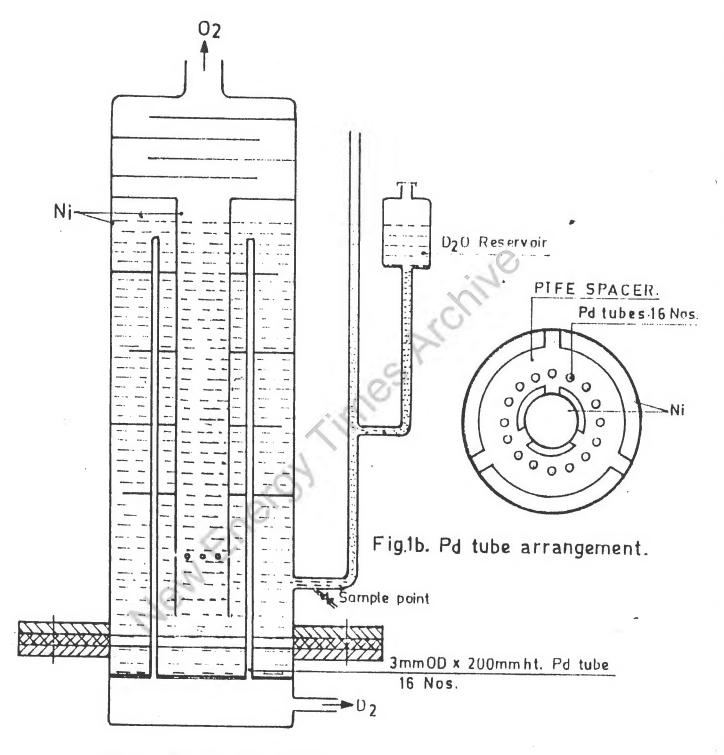


Fig.1a. Electrolytic Cell.

Electrolyser Systems used for Cold Fusion Studies

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The electrolytic cells and cell modules including complete electrolyser system were fabricated and provided by Desalination Division, Hydrogen Section, for the cold fusion studies carried out in collaborative work by Heavy Water Division, Neutron Physics Division and Desalination Division.

Two types of cell configurations were used in the studies. A single cell monopolar titanium electrode assembly and a bipolar multicell module consisting of Palladium – 25% silver alloy cathodes were fabricated.

Single cell titanium system

This cell consists of a tubular configuration with a central solid titanium rod forming the cathode and the concentric titanium outer tube functioning as anode. The current supply to the electrodes were provided by insulated flanged and connections which were also used to assemble the cell. The cell itself was housed inside a stainless steel tubular jacket with provision for electrolyte inlet and product gas outflow. The gas generated between the electrodes gives a bouyant force to keep the electrolyte in circulation so that an uniform temperature of the electrolyte could be maintained. The product gas mixture, consisting of Deuterium and oxygen, coming out of the cell was brunt in a burner condenser and the product heavy water formed was returned into the cell, thus forming an invariant closed circuit cell system. Two identical cells were provided so that one of the cell could be operated simultaneously with light water, generating hydrogen and served as a reference to the other cell operating with heavy water.

Multicell module assembly

A five cell module of bipolar filter press configuration having Palladium — Silver alloy as cathodes and porous nickel as anodes was fabricated using 0.1 mm thick 25% Ag—P4 alloy sheets. The electrodes were of circular geometry with an electrode area of 100 Cm². The cell module could be operated even upto a high current density of 1 A/Cm² and temperatures of 100° C. The module consists of 5 cells connected in series and the mixed gaseous product was carried out of the cell module by the recirculated electrolyte.

Electrolysis system

The two types of cell configurations are so constructed that they can be fixed into a circulating closed loop electrolyse unit for long term continuous operation. This will enable to study the accumulation of fusion products like tritium and helium after sufficiently long hours of continuous operation.

At integrated 5 cell module electrolyser system was fabricated and the same was operated on a continuous basis for detection of cold fusion Phenomena in Palladium matrix. The electrolyser consists of a closed loop wherein the product heavy water formed by burning of deuterium in the burner condenser could be recycled back to the electrolyser. Fig.1 gives a schematic flow diagram of the electrolyser system used in the cold fusion studies conducted in early experiments.

D.C. power is supplied to the cell module. The product gases viz. deuterium and oxygen, are carried along with the electrolyte ;to a vertical tube type gas—liquid separator. No separate circulating pump is used. The continuous generation of gases between the electrodes and the consequent gas lift is used for the circulation of the

electrolyte through the system. The hot electrolyte gas mixture is cooled in an electrolyte cooler to maintain a steady and desired operating temperature. Thermowells are provided before and after the cell module to measure the temperature rise across the module during operation. The electrolyte after separation of product gases returns to the cell module by gravity. The gas mixture leaving from the gas liquid separator is passed through a D2O scrubber to remove any NaOD carryover. It is again passed through a bubbler which also acts as a flash back arrester. The gas entering the burner-condenser is lit by an electrical spark mechanism and a spark detector is used for ensuring a steady and continuous flame at the burner tip. The burner condenser is a double walled jacketted cylindrical vessel which is cooled by passing chilled water through the annular space. Additional cooling coil is also provided inside the vessel to sufficiently cool and completely condense all the heavy water formed by burning of deuterium. The product Heavy water from the burner condenser is put back into the electrolyser to form a closed electrolyte loop. Heavy water can be added to the system through a separate feed pump for make up when the system is in open loop operation. Process parameter like temperature, pressure, cell voltage and cell current are continuously measured and indicated in the front panel of the system.

Electrolysis operation for cold fusion detection studies

The electrolyser system with 5 cell bipolar filter press module having pd-Ag cathodes and porous nickel anodes was operated on a continuous basis during May '89. The cell operating temperature was maintained around 60 C and pressure at about 0.6 to 1.0 kg/Cm². The applied current density varied from 0.4 A/Cm² to 0.75 A/Cm² with a cell voltage of 12-15 volts. The unit was operated for a total of 75 hrs. Periodic addition of heavy water was carried out as open loop operation was resorted to and samples of heavy water from the burner condenser taken out on daily basis for tritium detection.

Search for Electrochemically Catalysed Fusion of Deuterons in Metal Lattice

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Introduction

Recently, Fleischmann and Pons' have obtained evidence for the fusion of deuterons induced at low temperature, through the electrolysis of deuterium oxide at a palladium cathode. It is believed that this can be achieved if the deuterium loading of the lattice exceeds the value for PdD In view of the relevance of this problem to this Centre, a programme of work was initiated in this Division. This report is a summary of the behaviour of Pd and other metals during charging in alkaline heavy water.

Experimental

Materials, Reagents and Apparatus.

Electrodes of different metals were used. These include a hollow Pd cylinder (area 5.87 cm², thickness 0.4 mm), Pd ring (area .b 14.5Cm², thickness'2 mm), Pd foil (area 1.5 Cm², thickness 0.3 mm), Ti plate (area 8 cm², thickness 0.5 mm) and a triangular piece of Ni—Ti alloy of area 10 cm² and weight 10.5 gm. Platinum gauze, Pt disc or Pt foil of large area was used as the anode. Heavy water (99.87e purity) and pure Li metal were used to obtain 0.1 M LiOD. Iolar—2 N₂ or argOn was used for deoxygenation and stirring. All electrodes were lightly abraded with emery, rinsed with acetone and dried before use.

Electrolysis cells were made of quartz for the container and the lids were of PTFE or glass. Provisions and inlets for deoxygenation, addition and removal of solution and temperature measurement were made. Loss of D₂0 due to evaporation and due to evaporation and electrolysis was compensated.

Equipments:

Galvanostatic sources and current pulsing units were made in the laboratory. A Digital Panel Meter (PLA, DM-20), was used for voltage measurements. Neutron counting and tritium activity studies were carried out, as described later. Set—up is shown in Fig.1.

Results:

1. Differential Enthalpimetric studies

Twin cells composed of Pd as cathode (5.9 Cm²) and Pt as anode (gauze) in one cell was coupled in series with Pt as cathode (5.9 Cm²) and Pt as anode (gauze) in another cell. The purpose was to assess the magnitude of heat effects due to reactions like deuterium adsorption on the electrode, its recombination, dissolution in the lattice, etc. The cells were insulated by Dewar and 45 ml of 0.1 M LiOD were taken in quartz cells. The temperatures were monitored by matched thermistors using a bridge circuit and also measured with thermometers. The temperature of the two cells during charging is shown in Fig.2. The temperature of Pt—Pt cell remained nearly constant whereas the temperature of Pd—Pt cell increased and attained a high value. The temperature rise in Cell I (Pd—Pt) is due to absorption, dissolution and interaction of deuterons in Pd lattice.

2. Calorimetric Measurements of Enthalpy changes.

A modified isoperibol solution calorimeter with an accurate thermistor bridge was used. The cell with contents were enclosed in a tight-fitting Dewar and was immersed in a thermostat at 250C for thermal insulation. Electrodes of hollow cylindrical Pd

cathode(5.48 cm²) and Pt sheet anode with 50 ml 0.1 N LiOD were used. Iolar-2argon was used for deoxygenation and the solution was stirred mechanically. Charging was effected at different current densities and the terminal voltage in each case was noted. The solution temperature was monitored by the thermistor probe and bridge and also by a sensitive thermometer. The temperature increase as a function of time at different c.d. is shown in Fig. 3. The heat capacity of the cell and components was determined by electrical calibration and corrections for heat losses were applied on the basis of Newton's law of cooling (Fig.4). The results are included in Table 1.

3. Extended Electrolysis with Current Pulsing:

3.1 Cylinder Electrode: A hollow cylindrical Pd cathode (5.9 cm²) and a Pt gauze anode were used and 0.1 N LiOD was electrolysed in a quartz cell with nitrogen bubbling. Initially, a constant current of IA was used and when the temperature reached 600C pulsing was commenced between 1 to 2A at 1 s. interval. The temperature was controlled to 63°C by forced circulation of air. Neutron flux measurements were made and D20 was added for 2 was added for make up. After 41.8 hours during which 52.2 AH were consumed, the electrolysis was terminated. Measurement of tritium build-up in the final solution showed 3.75 μ Ci.

3.2 Ring Electrode:

(1) 65 ml of 0.1 N LiO,D in D20 were electrolysed in a quartz cell using a Pd-ring cathode (14.5 cm2) and two Pt discs as anode. At low current densities, the cell voltage was observed as a function of current and this is shown in Fig. 5. Later, electrolysis was carried out for4nine days with current pulsing between lA and 2A (80 h), 3A and 4A (20 h)and between 4A and 4.5 A (7 h) consuming a total of 296 Amp.hours. Neutron activity and capture 1; measurements were made throughout the duration of electrolysis. At the conclusion of the electrolysis, tritium content in the solution was found to be 16.25 μ Ci.

(2) The above experiment was repeated using 0.1 N NaOD and a total of 231Amp. hour were passed during 77 hours. Neutron counting and tritium activity measurements were

carried out.

(3) The same ring electrode, after degassing in vacuum, was subjected to 844amp. hour charging in 382 h. in 65 ml of deaerated 0.1 N LiOD. There was no significant tritium build-up in solution. The electrode was greyish black in colour.

3.3 Ni-Ti Electrode:

A Ni-Ti cathode (10 cm²) was used in 0.1 N LiOD and a total charge of 135 amp. hr were passed over 111 hrs. Appreciable neutron activity was observed. The cathode was observed to flake off and disintegrate in powder form.

3.4 Ti Electrode:

A Ti sheet cathode (8 Cm2 area) and Pt sheet anode were used in the electrolysis of 1 M NaCl in D₂0 at 34 mA.cm⁻². The terminal voltage was 4.4 V and temperature rose from 27°C to 36.60C in 53 minutes. Prolonged electrolysis did not show any significant increase in temperature.

4. Nuclear Measurements:

Four different types of measurements were made to identify the emission of

neutrons from the electrolysis cells.

1. Detection and direct measurement of neutrons were based on the use of He³ detectors arranged in a well-counter as well as a Li6 enriched scintillation detector (Bicron, Model NP-2). The detectors were calibrated by Am-Be source. Near complete rejection of high energy 1/-rays was ensured by proper discrimination of the 2.5 MeV Co60 sum peak using a Co⁶⁰ source.

2. Detection of high energy capture -rays of Gd,Pt and Pd was achieved by using Ge(Li)

or HPGe detectors.

3. Measurement of low energy capture —rays of energies 199, 944 and 1186 KeV was carried out with a HPGe detector. 4. Gross counting of 1/—rays of energy greater than 3 MeV was effected by means of a properly shielded 3" x 3" NaI (II) detector.

All the above measurements were done in combination to yield t-cross-validatied

results. However, in some cells, only a single type of measurement could be made.

Neutron counting was performed both in the MCS mode (using IBM-PG based 8 K MCA system) and in the PHA mode whenever possible. Different dwell times ranging from 0.5 sec. to 60 sec. were selected in the MCS mode to check whether neutron emission is continuous or in "bursts". The complete counting set up is given in Fig. 6. The results can be summarised as under:

a) Palladium hollow cylinder cathode (run 3.1)

The 1186 KeV gamma ray activity was measured every 100 sec. interval for more than 24 hours. In Fig.7, three definite 'spikes' can be identified. The duration of the 'bursts' was 14 to 20 minutes.

b) Palladium ring cell (run 3.2.1)A time correlated analysis of the neutron counts in the Li scintillation counter and measurement of r-rays of energy greater than 3 MeV in the NaI (Tl) detector were carried out (Fig.8). The correlation co-efficient for the 50

observations was 0.26 which is significant at the 5% level.

c) Ni-Ti cathode cell (run 3.3) Neutron counting was carried out using Hel as well as Li scintillation detectors with the former in both the MCS and integral modes and the latter in the MCS mode. The cell was run initially at lA current and when the current was increased to 2-2.5 A, three well-defined "spikes" were registered in the MCS mode as shown in Fig.9.

The count rates of He and Li scintillator are not correlated in any significant manner, possibly because of the different efficiencies of detectors and geometries.

However, tiny spikes are registered in the He detector.

d) Palladium ring cell (run 3.2.3)

Several spectra for the low energy capture gamma rays were detected using HPGe detector for durations from 10,000 to 50,000 seconds. In some spectra, 199, 944 and 1186 KeV capture could be identified though they were not traceable in other spectra. As the accumulated count rates for these energies are rather small, it appears that neutron emission is low and not continuous.

Discussion

Electrolysis of alkaline heavy water results in the splitting of D₂0 with evolution of deuterium at the cathode and oxygen at the anode. The electrode process involves charge transfer, adsorption of the intermediates, subsequent reaction and gas evolution(2) On pd electrode, the discharge reaction in alkaline solution is followed by a rate determining recombination of adsorbed deuterium atoms at low over potential, whereas at high over potential an electrochemical desorption reaction is favoured. In the case of Ni—Ti, the discharge reaction is always accompanied by rate determining electrochemical desorption reaction.

The uptake of deuterium by metals during charging depends on the reaction

$$MD_{ads} \longrightarrow MD_{abs} \longrightarrow MD_{lattice}$$
 (1)

and is, therefore, governed by the fraction of the electrode surface covered by adsorbed D, solubility of deuterium in the metal and its diffusivity. The observed rise in temperature of the electrolyte is not due to the exothermic dissolution of deuterium in Pd, Ni—Ti and Ti or due to other chemical factors.

The measured overpotential is composed of ohmic, activation, adsorption, diffusion and concentration overpotential terms. The ohmic resistance and polarisation resistance cause Joule heating and thereby contribute to observed changes in enthalpy. Had the cell systems been Q-reversible, then if the polarisation is reversed, the electrode reactions at Pd and Pt electrodes should also be reversed. In the upper limit the maximum Joule heating is the product of the cell voltage E and cell current i. In the case of Pd-Pt electrodes, the minimum voltage or back E.M.F for electrolysis of D₂0 was calculated from thermodynamic data as 1.54 V. Therefore, the electrical power available for Joule heating is (E-1.54)i and this is the lower limit. The enthalpy changes calculated for different c.d. and the ratio of thermal output to the power input, expressed as per cent breakeven are also included in Table 1.

The predominant process during d.c. polarisation is the electrolysis of D 0 and has been checked by measuring the volume of D_2 and 0. mixture 2 has been checked by measuring the volume of D_2 and 0. mixture liberated in a fixed electrolysis time using a precision integral flow-meter and by gas chromatographic analysis of the composition of the gasmixture. As excess heat is liberated over and above electrolysis, it is clear that some other reactions are responsible for the excess enthalpy observed. Pauling ' ascribed the excess heat to the formation of palladium deuteride. However, Bockris and coworkers. have shown that exothermic effects due to solution or D in Pd, recombination of D atoms, formation8 of D_2 0 etc.cannot account for the observed heat evolution. Normal chemical reactions cannot account for the generation of neutrons orthe production of tritium during charging Pd with deuterons. As has been pointed out by Fleischmann and Pons ' ', the results can be rationalised and understood on the basis of cold fusion reactions occurring between deuterons in the Pd lattice as indicated below:

$$_{1}^{2}D + _{1}^{2}D \longrightarrow _{1}^{3}H + _{1}^{1}H$$
 (2)

$$_{1}^{2}D + _{1}^{2}D \longrightarrow _{2}^{3}He + _{0}^{1}n$$
 (3)

In the present work, the emission of neutrons, reasonably above the background level, and the build up of significant tritium activity in excess of the blank value, have been confirmed in four different electrolysis experiments. In certain experiments neither the evidence for significant neutron emission nor any appreciable build up of tritium activity has been observed. It is likely that in such cases charging was not sufficient for ensuring optimum loading of the lattice with deuterons for inducing fusion. However, it appears that in addition to reaction channels (2) and (3), the possible occurrence of a non-emitting nuclear process cannot be precluded. This reaction can be written as

$$Pd + {}_{1}^{2}D + {}_{1}^{2}D \frac{lattice}{catalysed} > {}_{2}^{4}He + Pd^{*}$$

$$(4)$$

which implies that the lattice is excited to a higher energy level to conserve both momentum and energy. It is likely that during the subsequent lattice relaxation, the excess energy stored in the lattice is liberated as heat(5) This mechanism would lead to the formation and build up of He inside the metal and can possibly account for the observed low yield of neutrons or tritium in certain experiments. Accurate mass spectroscopic3/He4 ratio in the cathode material is needed to substantiate analysis of He/He4 ratio in the cathode material is needed to substantiate9this view. In conclusion, it is necessary to examine in detail the different parameters and optimise important factors like metallurgical history and pre treatment of the cathode, solution chemistry, surface chemistry and electrochemistry to achieve reproducible fusion

throughelectrochemical charging of metals in heavy water.

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Authors are grateful Lo Dr.R.M.Iyer, Director, Chemical Group for his keen interest and encouragement. Acknowledgements are due to Shri T.S.Murthy, Director, Isotope Group for kindly providing the data on tritium analysis. Support from Shri S.P.Chaganty of Electronics Division for scintillation detector and associated electronics facility, Shri J.D.Gupta and Ms Suman Kumari of Computer Division for the 8KPC—based MCA system and Dr. M.R.Iyer of Health Physics Division for 3well—counter and a MCS mode acquisition set up are gratefully He³ well—counter and a MCS mode acquisition set up are gratefully acknowledged. In particular, we wish to acknowledge the painstaking efforts and support of the following scientists of the Division in providing the reliable data cited in this work.

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2. Nuclear and electrochemical instrumentation — Ss. K.C.Thomas, J.R.Kale, R.G.Dalavi and A.W.Sahani.

3. Calorimetric measurements - Shri S.S.Sawant.

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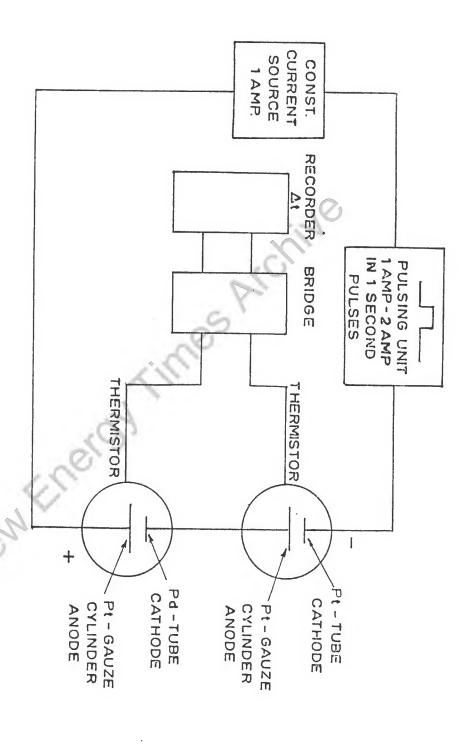
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Table 1

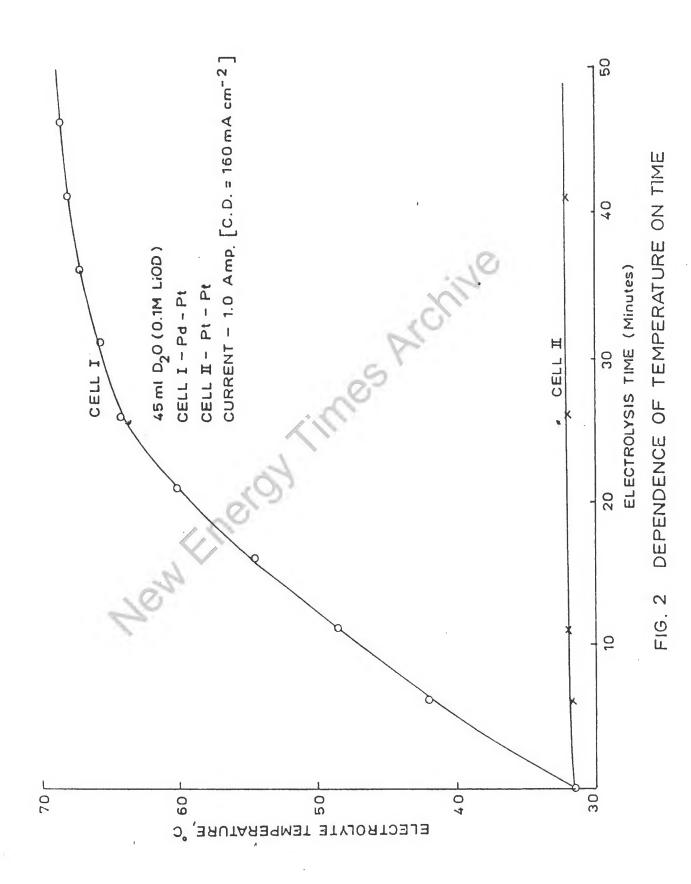
Excess enthalpy generated during electrolysis of LiOD Area of Pd hollow cylinder : 5.48 cm², Temp . 25°C.

Current density	Energy input	ΔH, measured	Excess enthalpy	Excess enthalpy (in % breakeven)
	JE Watts	Watts	Δ H measured $(E-1.54)$ i $\times 100$	△AH measured ×100
		3		
36	1.25	1.00	105	80
73	3.96	3.13	76	79
110	7.30	5.60	88	77
146	10.47	6.95	75	99
183	15.46	10.79	78	70
			10	



1361

FIG. 1 EXPERIMENTAL SET-UP



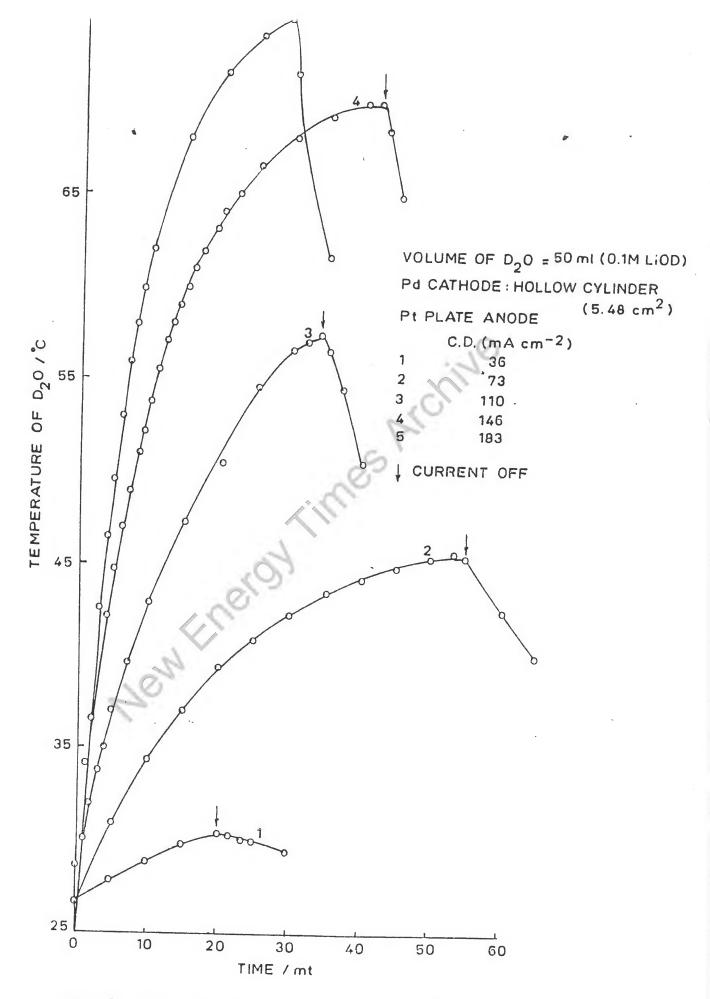


FIG. 3 TEMPERATURE CHANGES DURING CHARGING

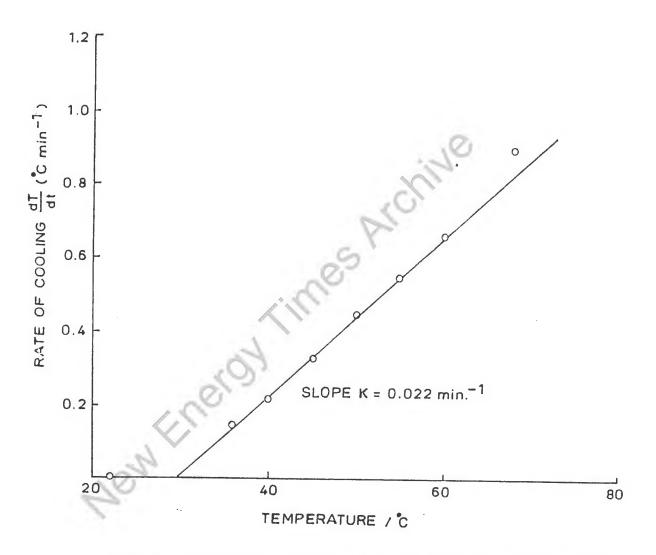
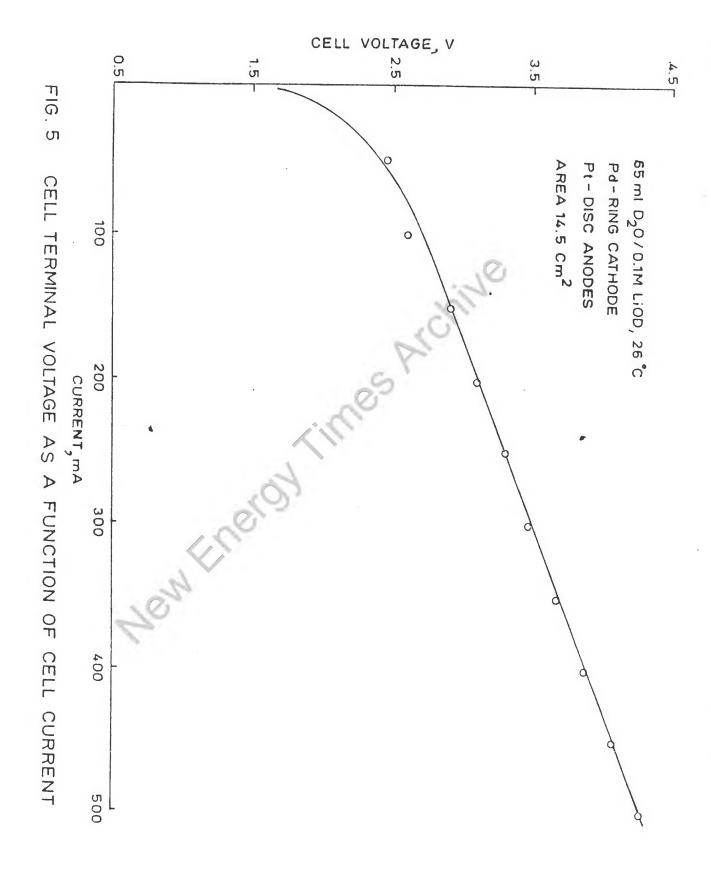
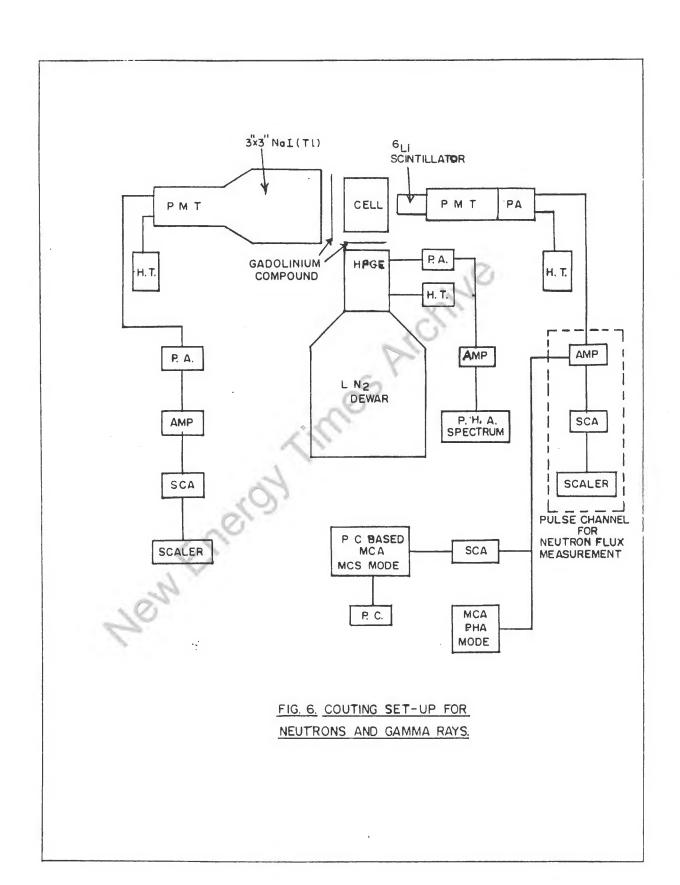


FIG. 4 TEMPERATURE-RATE OF COOLING, RELATIONSHIP





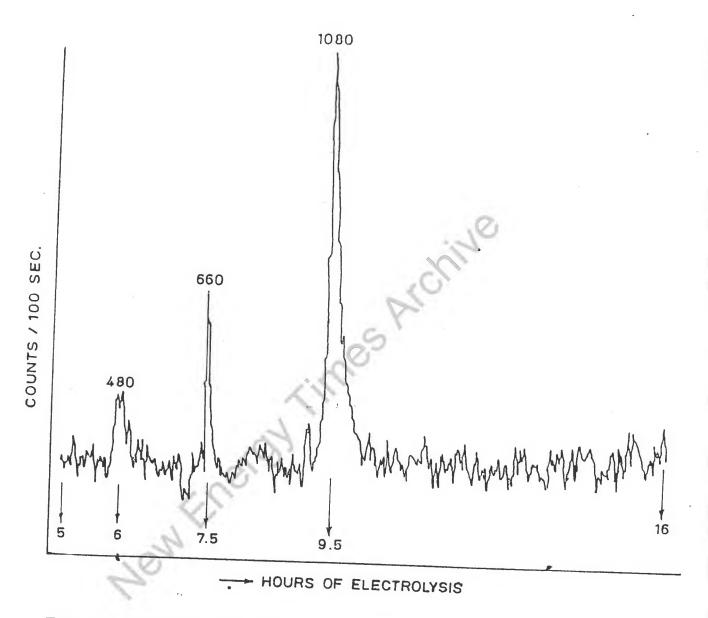
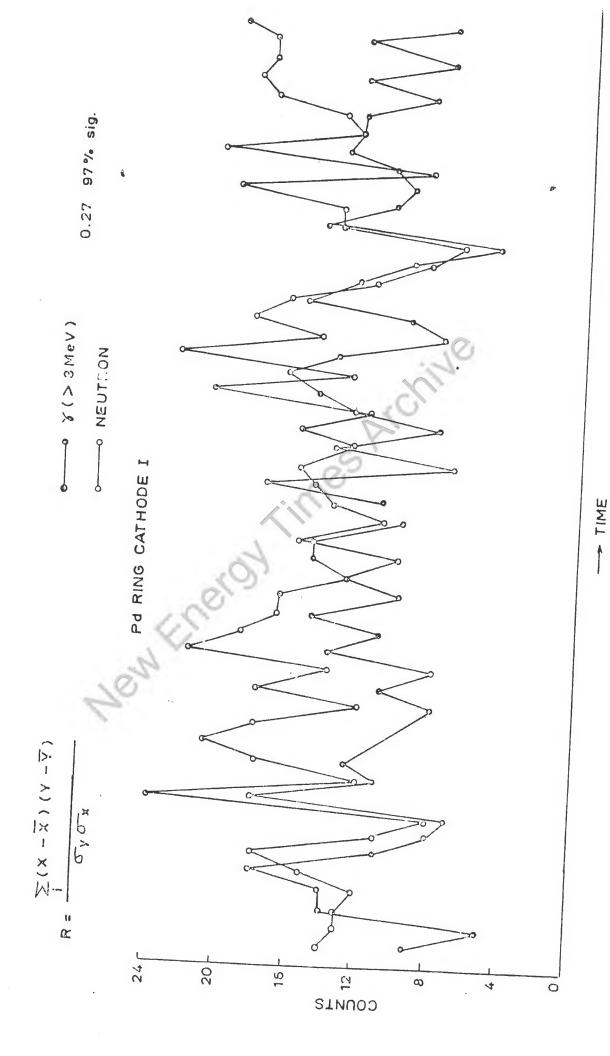


FIG. 7 COUNTS OF 1186 keV CAPTURE Y-RAY OF GADOLINIUM NoI(TI) DETECTOR



TIME CORRELATION OF NEUTRON AND GAMMA RAY ACTIVITIES F16.8

DWELL TIME 2 min.

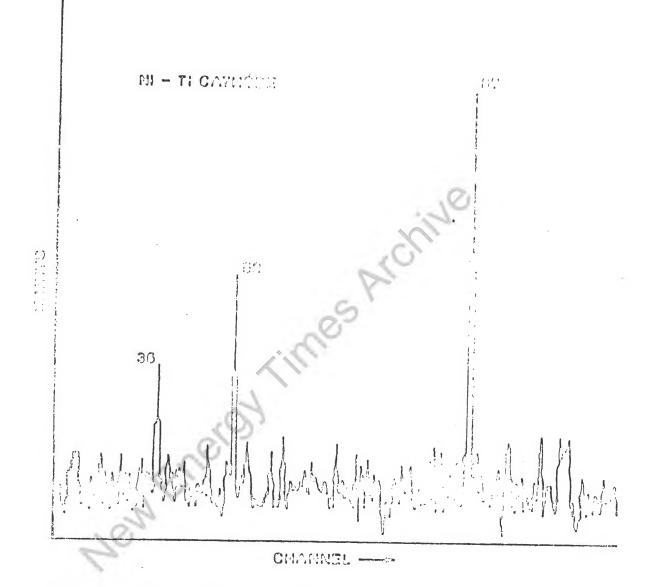


FIG. 9 DISTRIBUTION OF MEUTICON COUNTS
(MODE 50 SEC. DWELL WICE. LE CONTILLATION
DEVICTOR

BURST NEUTRON EMISSION AND TRITIUM GENERATION FROM PALLADIUM. CATHODE ELECTROLYTICALLY LOADED WITH DEUTERIUM

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Introduction:

Recently there have been many reports on the observation of neutrons, tritium and excess heat output from palladium and titanium cathodes electrolytically charged with deuterium¹⁻⁴. Steady neutron count rate observed during the operation of the electrolytic cells containing these cathodes and platinum anodes were in some cases only 1.5 to 3 times the back— ground rate³, whereas in some other cases a much higher steady emission rate (10⁴s⁻¹) was observed. In some experiments conducted at BARC earlier neutron bursts of two orders of magnitude larger than the background have been reported from electrolytic cells⁵. The possible nuclear reaction responsible for the emission of neutrons has been inferred to be the 'cold fusion' of D atoms (existing as D+) in the metallic lattice of the cathode

$$_{1}D^{2} + _{1}D^{2} \longrightarrow [_{2}He^{4}]^{*} \longrightarrow _{2}He^{3} + _{0}n^{1}$$
 (1)

We report here a large (larger than hitherto reported) burst emission of neutrons (signal/background ratio as high as 2000) from a thin ring shaped Pd cathode during the electrolysis of heavy water at relatively low cell currents and also tritium generation as measured in the electrolyte as well as in the water reformed from the sorbed gas recovered from the cathode.

Experimental:

The electrolytic cell design was optimized mainly with respect to observation of fusion products rather than the accurate measurement of excess heat output. This is because the quantity of heat required to raise the temperature of 1 g of D2O even by 1 K demands the occurrence of about 1013 fusions (see Discussion sec.) and hence heat measurement appears to be an in-sensitive method of confirming whether cold fusion occurs in the cathode or not. Fig.1 shows the schematic of the experimental set up. The electrolytic vessel (vol. ~250 cm³) used was made from quartz with a gas tight nylon cap. The cap had a number of penetrations for inserting electrode lead wires, thermocouple, reference electrode, purge gas inlet and outlet tubes. These tubes were made of 'Corning' glass. The Pd Cathode used was in the form of a hollow ring 2.5 cm in diameter, 1 cm in height and about 0.1 cm in thickness. The bulk density of the cathode was determined to be 12 g cm⁻³. The cell configuration was such that the ring cathode could be charged with deuterium from both sides as it was surrounded from inside and outside by Pt gauze anodes. The anodes were loosely sandwiched between pairs of Nafion membrane so as not to allow oxygen evolved at the anode to diffuse to the cathode surface. The electrolyte was D₂O of 99.86%2 of isotopic purity containing 0.1 mol dm⁻³ LiOD and was kept under constant circulation at a flow rate of 10 cm³m⁻¹ using a peristaltic pump. Incorporation of a heat exchanger in the circulation path along with a thermostated water bath (273-373 K) served to maintain the temperature of the electrolyte (and hence the electrode) at any desired value in this range. Nitrogen gas bubbling through the cell was done to reduce the dissolved oxygen level in the electrolyte. A saturated calomel electrode along with a luggin probe containing 0.1 mol dm⁻³ LiOD/D₂O dipped in the electrolyte was used as a

reference electrode to monitor the single electrode potential of the cathode.

A well type neutron detector assembly containing 24 numbers of ³He detectors embedded in paraffin filled to sufficient thickness and held between aluminum cylinders for thermalisation of the neutrons was used for n detection (Fig.2a). This configuration gave 8.6% efficiency for n detection. The signals were fed in parallel to a scaler with pre-set time and to a personal computer operated in a,8K multichannel scaling (MCS)mode with dwell time of 40 seconds per channel (Fig.2b). The latter was specifically aimed at detecting any Small burst release of neutrons which can otherwise get averaged out in a longer time duration pre-set time counting. Any pickup of extraneous signals like those emanating from the operation of pump motors, drilling machines, tesla coils and fluorescent lamps going off and on was thoroughly checked and the counter was found to have good stability and was immune to such electrical disturbances. A constant background of about 1.7 counts / s was registered without the electrolytic cell in operation for a duration of 10 days before setting up the electrolytic cell inside the counter well and also during times when the electrolysis was continuing but no burst emission of neutrons occurred from the cell.

Accumulation of tritium in the electrolyte was monitored by withdrawing periodically 1 cm3 samples from the cell at about 6 days intervals and measuring the T content using gel liquid scintillation "cocktail" and a Packard counting system. The counting efficiency was about 25%. The chemiluminescence effect due to the presence of LiOD was seen in the samples and counting was done till such effects died down completely and stable counting rates were obtained. The electrolytic gases from the cell were recombined over a Pt/polyester fabric catalyst and collected in a cold trap, and the resulting D₂O was checked for the presence of tritium. Loss of D₂O due to electrolysis and evaporation was made up by periodic addition of pure D₂O while losses due to samples drawn from the cell for tritium analysis were made up by adding 0.1 mol dm⁻³ LiOD solution in D₂O. The D₂O used for the experiment and for replenishments was from a single stock of heavy water. Temperature of the electrolyte was monitored by a chromel—alumel thermocouple encased in a glass tube dipping in the solution.

After the completion of the electrolysis run the Pd cathode was disassembled from the cell and connecting leads and dried. The absorbed gases from the cathode were recovered by heating it at 680 K in an evacuated chamber. From the pressure volume relationship and gas Chromatographic analysis of the deuterium content the volume of absorbed D_2 gas was computed to be $320~\rm cm^3$ at STP. This gas was then equilibrated over CuO at 680 K till there was no further reduction in volume and the water formed <-0.3 cm³) was collected in a thimble cooled to liquid N_2 temperature and this water also was counted for T.

Results:

Figs.3 and 4 show the burst neutron emission seen from the cell. While Fig.3 shows counts obtained in the MCS mode with time, Fig.4 shows the Count rate variation with time computed from the preset time <6000 sec.) scaler counts. Upto 14 days of electrolysis (charge passed ~2.24x106 Coulombs) only background neutron counts were observed and again after the 17th day of electrolysis (charge passed ~2.5x106 Coulombs) till the end of the run i.e., 32nd day (total charge passed ~3.65x106 Coulombs) only background counts were seen. During the burst Period <15th-17th day of electrolysis), initially a number of intense bursts were observed, each separated from the other approximately by. 2 hrs of quiescent period (Fig.3). Table 1 shows the actual duration of burst and the duration of the following quiescent periods. After about 7 such bursts the burst intensity decreased but bursts became more frequent so much so that in the later stages they appeared to be continuous.

The maximum burst signal to background ratio was ~2000 (Fig.4). In terms of absolute cOunts this corresponds to counts as high as 142000 in the 40 s time channel as against 5D-70 counts registered for the background (Fig.3). The area under the curve 11.1 Fig.4 gave an integrated neutron counts of 107 Using the 8.6% efficiency of the counter this works out to 1.2×10^{10} neutrons emitted from the cell in a period of about 72 h or a pseudo average 1, neutron emission rate of 4.6×10^2 n s⁻¹ (background average rate is1.7 n s⁻¹).

The electrolyte temperature was varied in the initial stages of the run by changing the applied voltage to the cell or by changing the set temperature of the thermostatic water bath. Ten hours before the burst emission and during the burst emission, temperature of the electrolyte was not altered and it remained in between 296 K and 300 K. Electrolyte temperature at any time of electrolysis remained above 293 K.

Tritium counting of samples (Table-2) drawn from the 5th-17 th days after the burst neutron emission (sample Nos. 4,5 and 6 in Table 2) showed an increase from 0.4 to 1.3 Bq.cm³ Low level counting methods (60 minute count duration) coupled with a stable system background yielded a standard deviation of only 3% on the count rates. The cell electrolyte volume being 250 cm³ an increase of 0.4 to 1.3 Bq. cm³ in tritium activity shows an extra input of T to the extent of 100 to 325Bq., amounting to 5.6—18.0 x 10¹⁰ extra tritium atoms accumulating in the electrolyte probably as D₂O. It is known that at temperatures above 293 K, electrolysis of D₂O does not result in preferential enrichment of tritium in the electrolyte⁵. In the present experiment electrolytic gases recombined over a catalyst and counted for tritium have shown tritium escaping from the cell. Hence the observed excess of tritium activity points to an extra source of tritium on the conservative side, i.e., not accounting for the tritium that is carried away in the gas stream during the electrolysis.

Degassing of the Pd cathode at 680 K at the end of the experiment yielded 320 cm³ of D₂ gas at STP. By reaction of this gas over CuO turnings at 680 K and quantitatively collecting the reformed water and, counting for tritium has shown 5.4 Bq. ($3x10^9$ atoms of T) accumulating in the Pd cathode.

Fig.4 also indicates the electrode potential as observed against a saturated calomel electrode. A rest potential of -1.0 V indicates that the electrode at 0.1 mol dm -3 alkali concentration behaves as a near hydrogen electrode. Monitoring potentials some hours into the burst emission period revealed fluctuations in the working electrode potentials. A shift to more positive value (by as much as 0.7 Volts in the cell under "OFF" and by 3 Volts in the "ON" conditions) was observed when burst emission occurred. Thus it looks as if there is depletion of deuterium gas at the surface of the electrode when burst emission of neutrons takes place.

Discussion:

Since the fusion of two deuterium atoms under the ambient temperature conditions is assumed to be induced by quantum mechanical tunneling, its probability depends on the nearest neighbor distance between the D atoms. The equilibrium distance between D atoms in D₂ gas being 0.74 A. recent experimental results(1-5) showed that in order to explain the observed rates of reaction the equilibrium distance between D atoms must be greatly reduced. Sun and Tomorak⁶ point out that at very high deuterium concentrations in the bulk, corresponding to a hypothetical PdD₂ crystal, the equilibrium distance between the two D atoms is increased by 0.2 A from the gas phase value of 0.74 A. They conclude that because of such large internuclear distance cold fusion of deuterium is very improbable inside a palladium matrix. But the question that remains unanswered is whether D+...ion in view of its high mobility in Pd matrix can encounter a significant

number of close collisions and in that process result in fusion reactions giving rise to neutron and tritium as fusion products from reactions such as

$$_{1}^{1}D^{2} + _{1}D^{2} \longrightarrow _{2}^{1}HE^{3}(0.8.2MeV) + _{0}n^{1}(2.45MeV)$$
 $_{1}^{1}D^{2} + _{1}^{1}D^{2} \longrightarrow _{1}^{1}T^{3}(1.01MeV) + _{1}^{1}H^{1}(3.02MeV)$
(1)

The present study has shown that for identifying whether cold fusion occurs or not, monitoring neutrons is a much more sensitive method than monitoring tritium, especially when the fusion events generating tritium are not very high . This is because of the Small value of the decay constant of tritium which requires an accumulation of 109 atoms to register 1 Bq. of tritium activity. In the present case although the neutron emitting events computed from the integrated neutron counts amounted to only 108 as compared to 1011 events computed from tritium data, the neutron emission occurred with big spikes and bursts. As against this, 1011 events through the tritium channel were inferred from an increase of 1.3 B9./cm3 extra tritium activity observed at the end of the experiment. Since the volume of the electrolyte used was 250 cm³ the above tritium concentration amounts to about 325 Bq.of tritium activity (1.8 x 1011 T atoms) held in the electrolyte. In contrast with the total number of T atoms released to the electrolyte, it was found that only 5.4 Bq of tritium activity (3 x 10° T atoms) were held in the pd cathode. But even this 5.4 Bq of T held in the cathode is 7 times more than what one would have obtained even if one takes into account back diffusion of tritium from the electrolyte to the Pd electrode. Since electrolysis of D2O at > 293 K does not result in T enrichment in the electrolyte, this back diffusion can only result in the the same T activity concentration in the reformed water, obtained by degassing the absorbed gases in the cathode and recombining over a catalyst, as is present in the original D₂O taken in the cell. The diffusion of T from electrolyte to Pd cathode would have given rise to a maximum of 0.72 Bq of T activity built up in the cathode over the duration of this experiment. But the actually observed activity of 5,4 Bq Shows that extra T has got generated in the Pd cathode. But it 100k5 as if the generated T most)y diffuses into the electrolyte rather than getting retained in the Pd matrix which is somewhat akin to n emission. Srom the variation in T activity observed at different times during the course of electrolysis (Table-2) it appears that T input into the electrolyte started after the hurst n emission occurred since the l6th day sample which was taken during the burst n emission registered the same T activity as the sample taken id the initial stages of the run. But during the T generation period (17th-32nd day) there was no n emission observed over the background rate.

From the volume of D₂ gas trapped inside the Pd electrode8 (320 cm³ at STP actually collected) the composition of Palladium-Deuteride that would have given rise to the burst n emission was inferred to be between PdD_{0.3-0.4} Using the known diffusion coefficient of D < lxlO-7 cm²/s) lt was computed that through the 1mm thick cathode used in this study, D atoms can enter and come out through the other side in about 30 hours time thus pointing to the attainment of at least PdDo. 25 configuration in about a day's time. But, as shown by D2 gas trapped in the cathode at the end of the experiment (32 days), only at the end of the 32nd days this composition seems to have been reached. The reason for this could be the blackish loose coating forming on the Pd Cathode during the course Of the experiment which perhaps acted as a diffusion barrier for the entry of D atoms. EDXF analysis of this coating revealed it to be platinum which might have arisen from anodic dissolution of pt at the applied cell voltages. (Which were higher than the reversible pt/pt2+ potential). The reasons for not sustaining the burst neutron emission for more than three days could be summarized as follows: Those sites in Pd matrix in which fusion of D atoms occurred might have been the really active site@ and once D atoms got depleted from those sites further replenishment did not take place possibly either because of the barrier coating which only favoured molecular D2 formation

from the cathode surface with no further entry of D atoms into the matrix or because such sites had turned amorphous due to the intense local heating that might have occurred from the deposited kinetic energy of the fusion products.

The literature reports on cold fusion describing the large heat output from Pd electrodes of different configurations is rather confusing. In general very few groups have been able to reproduce the large heat output reported by Fleischmann et all and Mathews et a1(4) II@ our laboratory we have carried Out several runs on electrolytic cells using Pd cathodes of various shapes and dimensions (including a solid cylinder 1 cm diameter and 1 cm long) and pt anodes in 0.1 mol dm⁻³ LiOD/D₂O along with control runs using both electrodes of Pt in the same electrolyte as well as Pd cathodes and Pt anodes in 0.1 mol dm-3 LiOH/II2O and were not able to observe any excess heat output within the limits 0\$ uncertainty of our calorimetry, Which is estimated to be about ± 10%. In fact, by comparison with experiments where the cell contents were heated by a kanthal wire heater, we found that in "both control and actual experiments, the observed heat output from the electrolytic cell (under steady Conditions) was the same as the electrical power input (VXI), making one wonder, as done by Santhanam et al(3) as to where from the power required to sustain the electrolysis comes. The answer must obviously be that such comparisons, even after taking precautions to stir the system are subjected to various sources of error, and in our view very precise and unambiguous electrolytic calorimetric experiments which can detect heat output corresponding to steady excess temperature of 0.1 - 1 K are yet to be reported. Even to match a cooling rate of 0.1 K per minute in a cell of dimensions reported in the present paper it would require the occurrence of fusions at a steady rate of about 1010/s. As discussed above, the observations of n bursts revealed that the occurrence of fusions is not at a steady rate of the desired magnitude and hence there is no wonder that the excess heat output went undetected.

No significant heat generation was observed in the present study and heat generated in cold fusion if any, could not be differentiated from the background Joule heating. Considering the total number of fusion events that occurred and the heat capacity of the system it is not surprising that no significant temperature rise was observed.

Conclusions:

The present study has shown evidence for cold fusion phenomenon in an electrolytically charged Pd matrix in terms of neutron and tritium as the signatures. The tritium channel seems to be favoured over the neutron channel. During the period of our experiment a total of 10¹¹ fusion events leading to tritium generation were observed whereas the neutron channel accounted for Only 10⁸ fusion events. No significant heat output over the Joule heating could be observed. The present study has also revealed that the effect is small and not a sustained one. Energy production from cold fusion of deuterium in an electrolytically charged Pd matrix in a sustained manner may require more systematic exploration to identify the various parameters governing the occurrence of the process, not the least important among which is the proper pretreatment of the electrode.

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 $\frac{Table-l}{\text{Duration of burst neutron emission and that of the intervening}}$ quiescent periods from the Pd-Pt electrolytic cell

Burst (h)	quiescent period (h)
2.6	
	0.25
2.1	2.4
2 0	40.2.4
2.0	3.3
1 0	5
1.0	2.8
0.2	
	1.2
0.3	
	. 1.1
0.05	
C.C.	0.5
	400

After the last quiescent period following the 7th burst almost continuous emission of neutrons at different rates was observed.

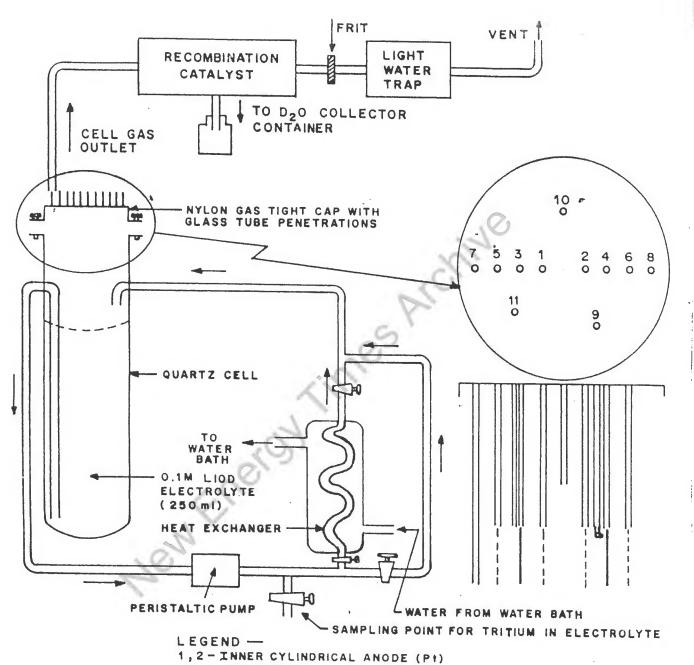
Table-2

Tritium content in the electrolyte (0.1M LiOD)

of Pd-Pt electrolysis cell

Canala N.	Duration of		itium activit
Sample No.	electrolysis	(cumulative)	(Bq/ml) *
	(d)	$(\times 10^{-6})$	
		,C	and then the two two two time day and then we were
1	0	0	3.65
2	7	1.07	3.32
3	16	2.36	3.65
4	20	2.72	4.94
5	26	3.21	4.24
6	32	3.55	5.2

^{*} standard deviation : \pm 3 %



3,4 - CATHODE RING (Pd)

5,6 - OUTER CYLINDRICAL ANODE (Pt)

7 - GAS INLET

8 - GAS OUTLET

9 - LUGGIN PROBE FOR CALOMEL ELECTRODE

10 - Cr - AI THERMOCOUPLE

11 - D20 REPLENISHMENT INLET

FIG. 1 EXPERIMENTAL ASSEMBLY - SCHEMATIC.

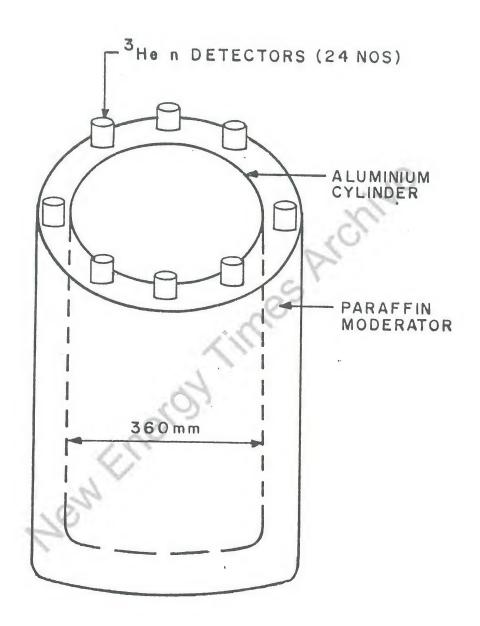


FIG. 2a WELL-TYPE n DETECTOR

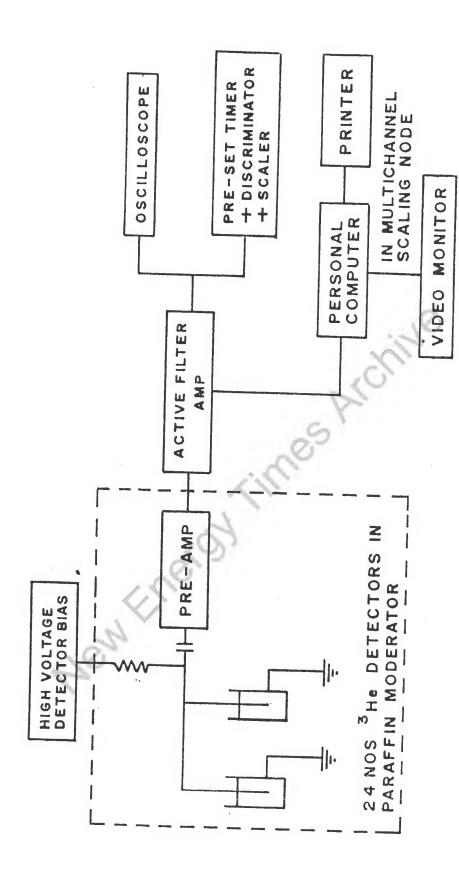
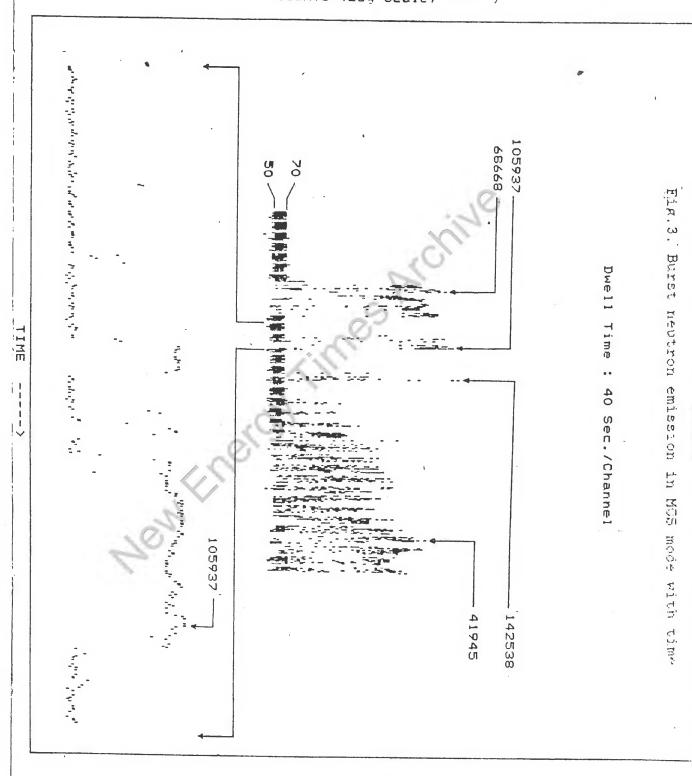
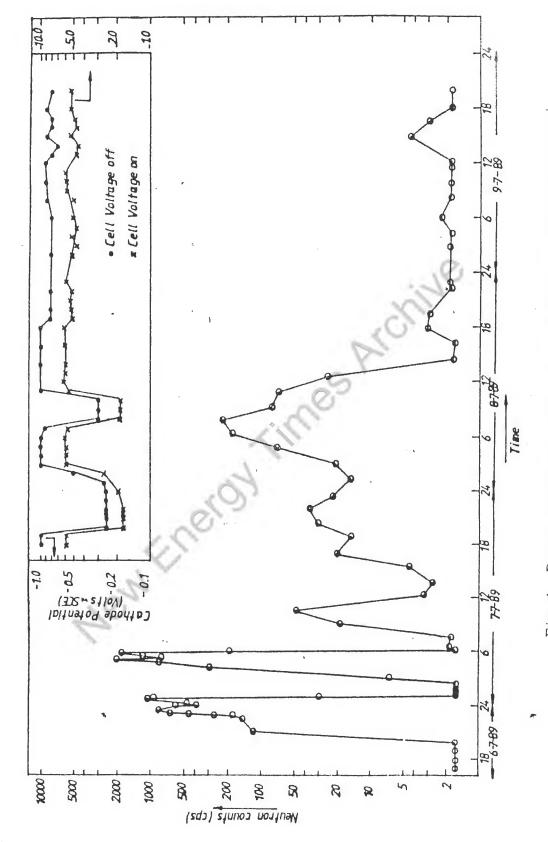


FIG. 2b SETUP OF NEUTRON COUNTER.





Burst neutron emission rate and Cathode Fotential variation with time Fig. 4.

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Search for Helium in the electrolysis of D₂0 with Palladium Cathode

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In nuclear fusion reactions involving D2 helium may be one of the possible products. During@ electrolytic dissociation of D20 with Platinum/Palladium electrodes if any fusion reaction (1,2) is taking place via the helium pathway with helium escaping to the gas phase it should be possible to detect and estimate the yield quantitatively. Helium is reported to have been observed in excess of the background in the electrolysis experiments conducted at the University of Utah(1) The number reported is of the order of 1012 atoms per second (-0.013 ul per hour). In presence of large excess of D₂ and O₂ generated during electrolysis detection of He3/He4 in the gas phase poses many problems. Among the methods available for the detection of low amounts of helium gas chromatography (GC) and mass spectrometry (MS) are normally preferred. However in this case mass spectrometry (MS) is complicated since helium is in trace concentration and large excess of D₂ will interfere with the signal due to He³ or He⁴ ions unless fractional mass difference method is adopted. In order to concentrate the helium in the gas phase an experimental technique has been devised wherein D2 and O2 generated by electrolysis is catalytically recombined in situ facilitating gas collection over long periods of electrolysis. The results obtained by this technique followed by gas chromatographic analysis are given in this paper. Though GC/MS analysis of the enriched sample may be more conclusive, a suitable GC/MS gas inlet interface compatible with D₂/He is difficult to get.

Experimental:

The electrolysis cell and the gas manipulation system is shown schematically in figure 1. This consists of a glass cell carrying a ground glass joint wit)? the cap connected to a mercury manometer with a 20 ml expansion bulb at the bottom, a silicone septum carrying vacuum tight electrode leads and a vacuum stopcock connecting the cell to a modified Toeppler pump with facilities to measure the gas pressure, volume and also to pressurize and transfer the collected @ae to a syfin@e salnplil3@lnanifold through a three way stop cock(3). The third limb of this stop cock is connected to rotary vacuum pump. The catalyst for recombination of D2 and O2 at room temperature is a specially prepared platinum catalyst deposited on a thick synthetic fabric. It is freely suspended on the top inner side of the electrolysis cell. The catalyst has been independently assessed for H₂/O₂ recombination efficiency and found to have T₁/₂ for H₂ reaction of less than 15 seconds in presence of sufficient 02 with gas volumes upto about 500 ml. The gaseous products are analysed by gas-chromatography using thermal conductivity detector either (1) or) a 5meter x 3 mm id molecular sieve 5A Column at 250c with argon carrier gas which gave clear separation between helium and hydrogen for the analysis of trace amounts of helium in the sample or (2) on a 2meter x 4 mm id. molecular sieve 5A column-at 25 with with helium carrier gas for the analysis of H2(Or D2)' 02and N2 in the sample. The lowest almost of helium detectable in the presence of large excess of hydrogen under the experimental conditions is about 0.01 ul (or about 1 ml sample with 10 ppm He). The second column provided a reasonably good analysis for the composition of the residual gas with respect to major constituents. Total volume of the electrolysis cell and the gas manipulation system as well as the volume of each segment of the system has been determined;d to facilitate computation of the gas volumes and composition at any stage of the experiment. The electrolysis experiment has been carried out in three stages. In these experiments 20 ml of 0.1 M LiOH or LiOD is used as the electrolyte. Experiments I and II are carried out with stainlesssteel Cathode and Anode mounted in concentric tubular

configuration with 1 lnm electrode spacing. The electrodes are approximately 1.5 cm X 3 cm X 0.04 Cm in size. These experiments are used to test the system with respect to leak tightness, gas recombination efficiency, trace helium recovery and analysis. Experiments III to VI are carried out with palladium/platinum electrodes. In this case a palladium pl"ate (1 cm X 1.5 cm X 0.15 cm) is sandwiched between two platinum plates (1 cm X 1.5 cm X 0.05 cm).

Prior to starting electrolysis the entire system including the cell with 20 ml electrolyte is evacuated through the threeway stop cock to thoroughly degas the electrolyte. After attaining vacuum the cell is isolated from the pump and rest of the system and the electrolysis started and continued for the required duration by connecting the electrodes to a D.C. power supply. At the end of the electrolysis the power is put off, system is allowed to stabilize till no further change in the mercury level of the manometer is noticeable. At this stage by suitable manipulation of the three stop cocks and mercury reservoir gas transferring, compression, measurement of pressure and volume and sampling for analysis are carried out. Whenever necessary desired reactant gas is introduced into the electrolysis cell through the silicone septum carrying the electrodes by means of a syringe. The electrolysis cell isolated under vacuum is found to retain vacuum for more than 48 hours (drop in mercury level — 0.5 cm). The results of the experiments with relevant details are given in Table 1.

Conclusions:

- 1) In contrast to the stainless steel electrodes Palladium cathode/Palladium anode configuration resulted in D₂ deficient gas composition and Palladium Cathode/Palladium anode configuration showed 0₂ deficient gas. In the initial stages of electrolysis much of the hydrogen (or D₂) may be absorbed by palladium reaching equilibrium in about 2 1/2 hours as indicated by the steady pressure build up.
- 2) Though all the released gas is contained and concentrated to residual 1 to 3 ml Volume no helium could be detected in all the experiments.
- 3) Hydrogen (or D₂) absorbed on the electrode is slowly released under vacuum Platinum catalyst can be incorporated in the cell design to recombine the evolved gas thus facilitating D₂0 recovery and also possible recovery of any gas phase back into the aqueous phase.5) In case an independent proof, for the cold fusion via the helium pathway is available, this method can be adopted suitably to detect helium and substantiate the finding with the possibility of unambiguous evidence obtainable from GC/MS analysis of the concentrated residual gas.

Explanatory note to the Table:

- (a) In case of I and II electrolysis is carried out for varying duration to ascertain the viability of the method and recombination efficiency of the catalyst.
- (a)* In these cases 150 ul of 1 % He in argon is added to the cell prior to electrolysis and the residual gas at the end analysed for He. Recovery is found to be better than 90%. (b)
- (b) (i) In case of III to VI initial evacuation of the system is carried out with the D.C. potential for about 5 minutes, then evacuation continued with the potential off for about 5 minutes, system isolated and the regular electrolysis carried out (system purging).
 (ii) In these cases mercury level in the manometer showed a steady drop for the first 2 1./2 hrs and then slowed down.
- (b)++ In case III the residual gas is used up in analysing for trace helium. Hence D2:02

composition is not determined.

C) At the end of experiment IV the electrolysis cell is maintained in the isolated mode overnight. The mercury level dropped by about 2 cm. overnight and the residual gas on analysis showed 90% D₂ indicating release of D₂ under vacuum from the electrode.

Acknowledgement:

The author is obliged to Dr. N.M. Gupta and A.D. Belapurkar of Chemistry Division for providing the platinum catalyst. The author is also thankful to Dr. R.M. Iyer, Director, Chemical Group and Dr. J.P. Mittal Head, Chemistry Division for their keen interest and helpful suggestions during the course of this work.

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Experiment	I (a)	II(a)	(S)	(q) ^{AI}	(9)^	(g)IA
Electrodes	Concentric Stainless Steel Cathode	Concentric Stainless Steel Cathode and Anode	Palladium Cathode Platinum	Palladium Cathode Flatinum Anode	Platinum Cathode Palladium Anode	Palladium Cathode Platinum Anode
Electrolyte	LiOH,0.1M in H ₂ O	LioD 0.1M in D20	LioD, 0.1M in D_2O	Liob, $0.1M$ in D_2O	Liob, 0.1M in D20	LioD,0.1M in D20
Electroly- sis Voltage Current and Duration	4.0 V -250 mA, 1h & 2h	4.0 V -250 mA 2h,2h,4h 1h,6h,6h	4.0 V ~250 mA 3h	5.0 V -500 mA 6h	4.5 V -450 mA 2h	4.0 V -27'5 mA 6h
Residual gas Volume after Electrolysis and Composi- tion(25°C/ 76 cm Hg)	-1 ml H2:02 -2:1	-1 to 3 ml D2:02 -2:1	26 ml -90% O ₂	76 ml ~90% O2	48 ml	28 ml -94% O ₂
Volume of reactant gas added	1 1	! !	50 ml H ₂	30 ml H ₂	24 ml O ₂	46 ml H ₂
Final gas volume and composition	-1 ml H2:02 -2:1	-1 to 3 ml D2:02 -2:1	-3 ml (++)	_1.6 ml D2:02 _1:5	-1.1 ml D2:02 -4:1	_5.6 ml D2:02 -1:8

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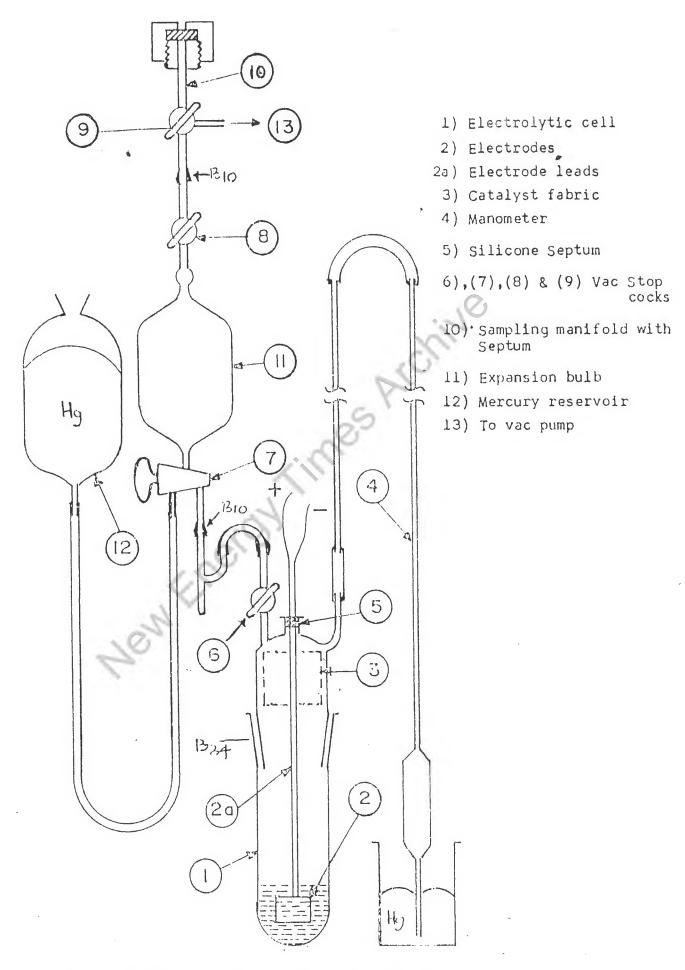


Fig. 1: Electrolysis cell and gas handling system.

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Microcalorimetric Investigations on the Differential Heat evolved in Electrolysis of H₂O and D₂O

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Microcalorimetric investigations were carried out to evaluate differential heat evolution in the electrolysis of D₂O and H₂O in presence of 0.1 M LiOD or LiOH as an electrolyte. A calvet type (Setaram, C-80) microcalorimeter was employed for this purpose. Two identical stainless steel cells (height 8 Cm, inner diameter 1.5 Cm) were equipped with similar Pd and Pt electrodes of 0.1 and 0.2 Cm.—diameter respectively the length of both the electrodes being 7 Cm. The differential heat evolution was measured for different current density values.

Prior to electrolysis experiments, the thermal balancing of the two cells was attained by adjusting the volumes of the D₂O and H₂O in the two cells. Both the cells were heated at the identical predetermined linear rate in the range of 0.2 and 20C min. It was observed that the best match between the two cells could be obtained by taking 11.1 ml of H₂O and 10 ml of D₂O in two cells. With these of liquids in two cells, the cell resistances were matched by adjusting the inter electrode distances. The electrolysis was then carried—out by connecting the two cells either in the series or in parallel to a stabilized power supply. The gases evolved during electrolysis were vented out through 30 Cm long stainless steel tube of 0.2 Cm inner diameter. The evolved gas flow rate was monitored periodically using a soap film flowmeter and was found to be dependent of current density. In a typical case when 101 ml H₂O and 10 ml D₂O were used in two microcalorimeter cells the flow rate of evolved gases under equilibrium condition of electrolysis (current density 176 mA/Cm⁻² Was found to be 1.9 ml min⁻¹ for H₂O cell and 1.6 ml min⁻¹ for D₂O cell. When the current density was raised to 352 mA Cm⁻² the flow. rates were 3.53 and 3.15 ml min⁻¹ for H₂O and D₂O cells respectively. A small discrepancy in flow rates of evolved gases from two cells was observed even when similar amount of H₂O was taken in both the cells.

The temperature of the microcalorimeter vessel was evaluated carefully and arise of about 0.20c for current density of 176 mA Cm⁻² and of 0.4°C for the current density of 352 mA Cm⁻² was observed.

The nature of the curves obtained in microcalorimetry data indicate that one of the cells gets heated more than the other during electrolysis. This difference could be attributed to several factors such as difference in rate of electrolysis, different rate of diffusion of H and D through the outlet tube and the difference in the heat capacity of H_2 and D_2 and O_2 gases evolved in electrolysis. No conclusive evidence is, however, obtained so far to suggest that a higher heat is evolved in the electrolysis of D_2O as compared to that of H_2O under identical test conditions. Experiments are being continued to evaluate the effect of different pretreatments to palladium electrode on electrolytic behavior of H_2O and D_2O .

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Cold Fusion Investigations at ROMG through Electrolysis of Heavy Water with Pd Cathode

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Part I: Initial Experiments (22nd April to 12th May 1989)

Introduction

Following a recent publication by Pons and Fleischman on Electrochemically Induced Nuclear Fusion of Deuterium /1/ experiments were initiated in our laboratory keeping the electrolysis conditions as close as possible to those of the authors. Due to non availability of a sensitive neutron counter, initial experiments were restricted to measurements of temperature changes occurring in the electrolytic cell to study the excess heating reported by the authors in the electrolysis of heavy water with palladium as the cathode. Tritium measurements in the heavy water following electrolysis were also made. Control experiments were carried out using platinum cathode in place of palladium. In this report, a brief description of the results obtained along with electrolysis data plots is given.

Experimental Details

The Cathode consisted of a palladium tube which was used earlier in gas analysis apparatus for hydrogen absorption. Prior to its use as a cathode, it was polished to remove surface film if any and it was heated to almost red hot condition to expel any occluded gases.

Details of palladium cathode are Length 6.0 cm; O.D 0.4 cm; Weight 4..2 g; Volume 0.36 cm³; Surface area 7.5 cm² Length of cathode dipping in electrolyte solution 3.0 cm Volume of dipped portion of the electrode 0.18 cm³; Surface area of dipped portion of the electrode 3.75 cm²; Weight of dipped portion of the electrode 2.1 g

The Platinum Cathode used in control experiments was a Platinum rod of 2 mm dia. was cleaned and dried before use. Length of platinum cathode dipping in solution was .03 cm. The Anode comprised of a gauze shaped into cylindrical form.

Electrolyte solution: Lithium hydroxide solution (0.1 M) with heavy water (isotopic purity > 99..86% W D₂O) as solvent.; Weight of the electrolyte solution taken for electrolysis: 86 g.; Voltage supply for electrolysis: Constant voltage supply unit with meters for voltage and current readings; Temperature of the electrolyte solution: This was read on the thermometer immersed in the solution.

Tritium analysis; Heavy water samples after electrolysis were sent elsewhere for tritium measurement.

Cell Assembly: Fig.1 shows the cell assembly and the associated set up. There was no stirrer or gas—sparing through the solution during electrolysis. It was assumed that the gases liberated during electrolysis would help in stirring the solution.

Discussion

In the preliminary experiments, voltage-current characteristics of the cell were

studied with varying anode geometry with a view to get high current density on palladium cathode. It was observed that current rose sharply with a wire—gauze anode in cylindrical form, surrounding the cathode. The variation of current with applied voltage is shown in Fig.2. Suitable geometry was then chosen so that there was no possibility of electrodes contacting each other during the course of the experiments.

With a regulated voltage supply unit, voltage remained constant in each experiment (except for small adjustments to keep the current constant in Expt. No.1). In experiments with lower applied voltages (3 – 3..5 V) current also remained constant. However, in experiments with higher voltages (4 V and above), current was found to increase as the electrolysis progressed, which could be partly due to higher temperature of the electrolyte solution.. Readings for temperature, voltage and current were taken at regular intervals. To establish whether the reported excess heating (over and above that due to Joule heating) is peculiar to Pd-D₂O system only, experiments were carried out in almost identical conditions for Pt-D₂O system where Pt and Pd are cathodes respectively, anode (Pt wire gauze) remaining the same in all the experiments.

The experimental details and some of the observed and calculated data are summarised in Table 1. Graphs have been plotted for all the experiments listed in Table 1 showing temperature changes against the input energy in W-minutes, obtained by cell current multiplied by (cell voltage — 1.54 V) and time in minutes. They are shown in Figs.3,4,5 and 6.

To calculate excess heating if any, in Pd-D₂O systems (Expts. No.1 and 3), accurate calorimetric measurements would be necessary, taking into consideration the heat lost to the surroundings. Our set up was not designed for such measurements. However, it was thought that comparison of the results obtained between Pd-D₂O and Pt-D₂O systems would help in establishing the excess heating observed by Pons and Fleishmann in their paper mentioned earlier. Experiments No. 2 and 4 were carried out for this purpose.

Thus comparing the results in Expt.1 with those in Expt. No. 2 for Pd-D₂O and Pt-D₂O systems respectively on the basis of similar rate of Joule-heating and other factors such as heavy water equivalent of the cell and its contents remaining almost identical, the T (i.e. the difference between the temperature at any given interval of time and initial temperature of the cell) observed in both the cases at any given W. minutes did not differ by more than a degree centigrade. This can be seen in Figs 3 and 4. The same data are superimposed in Fig.7 for better comparison. Under similar conditions, (Pd rod 0.2 x 10 cm; current density 64 mA/cm2) the excess rate of heating calculated from Table 1 in the paper of Pons and Fleischmann would give a value of 0.28 W for the Pd cathode in our experiment. This should give more than 60% additional heating in Pd-D₂O system as compared to that in Pt-D₂O system. This was not observed as can be seen in Fig.7.

Similar experiments (Expt. No. 3 and 4) were carried out at higher applied voltages and hence at higher current densities. According to the results of Pons and Fleishmann in Table 1 of their paper (Pd rod 0.2 x 10 cm), the excess rate of heating increased with current density. By taking the factors from their Table for the expected excess rate of heating, the calculated values for Expt. No.3 are 0.42 - 0.63 W which correspond to about 23 per cent additional heating in Pd-D₂O system as compared to that in Pt-D₂O system. Fig.5 and Fig.6 show the temperature profiles for Pd-D₂O and Pt-D₂O systems respectively and Fig.8 shows the superimposed plots. Here also, there is no indication of any excess heating in Pd-D₂O system.

Summarised data of Electrolysis Experiments
Anode: Platinum wire gauze as given in 2.2

Σ W-:Nin at 't' min		124		124	0	202	210			
		0 45	5	0.45		2.77	2.38			
	Watts range (W)		0+.0	0.43 - 0.45		1.73 - 2.77	1 78 - 2.38			
	on Final $\triangle T$ (V-1.54) it. temp. n) (°C) (°C) (V)		9.2 1.61 - 1.51 3.50 - 3.50	1,96		3.46	70 2			
	(°C)		9.5	77 00		22.1	-	71.6		
	Final temp.		37.9	0 10	. 0.70	50.3		49.8	Š	•
	Duration of Expt.		285		285	0		16		
	Current Density ₂	1	29		09		134 - 212	0 ~ 300		
\$	Current (A)		200	(7:0	0.22 - 0.23		0.5 - 0.8	0.45 - 0.63		
	Expr. Cathode D ₂ O Volts Current (V) (A)			3.15 - 5.57 0.27	3.5	1	5	7. 7.	,	
	D ₂ 0			D_2O		720	0,0	(D2C	
	Cathode			Pd-tube		Pt-rod	Pd-tube		Pt-rod	
	Expt.	o Z		-		7	3		4.	

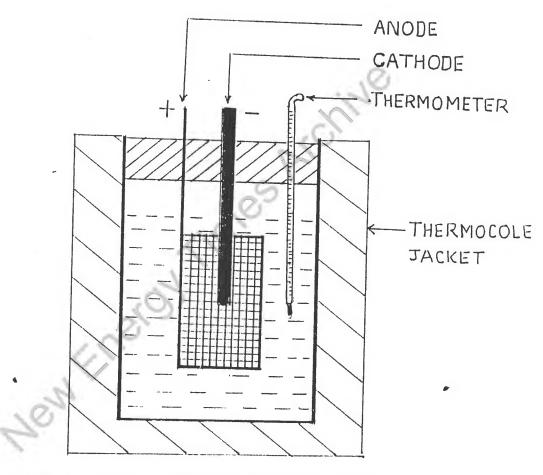


FIG. 1 CELL FOR ELECTROLYSIS

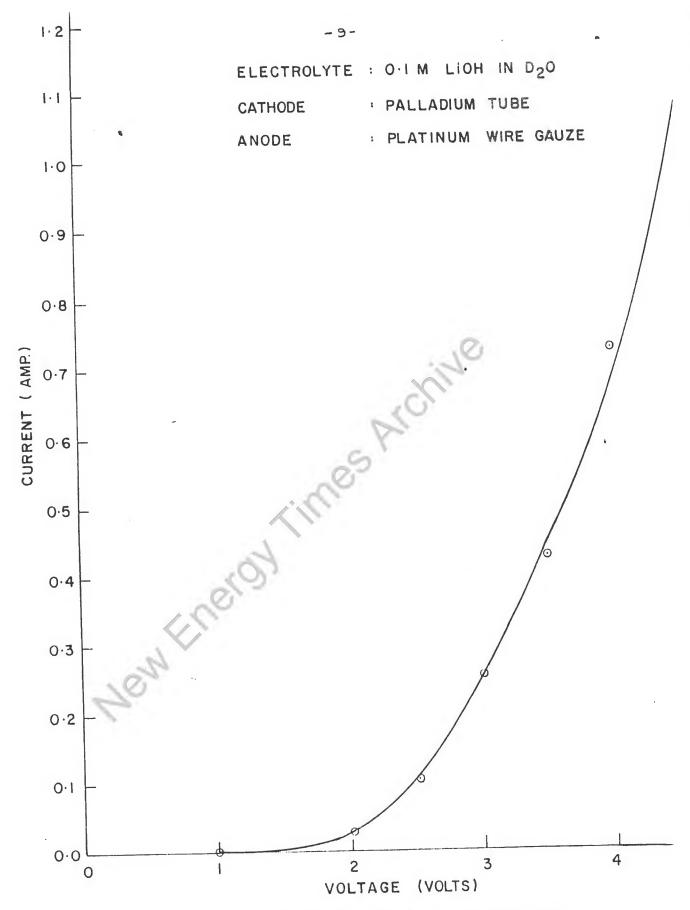
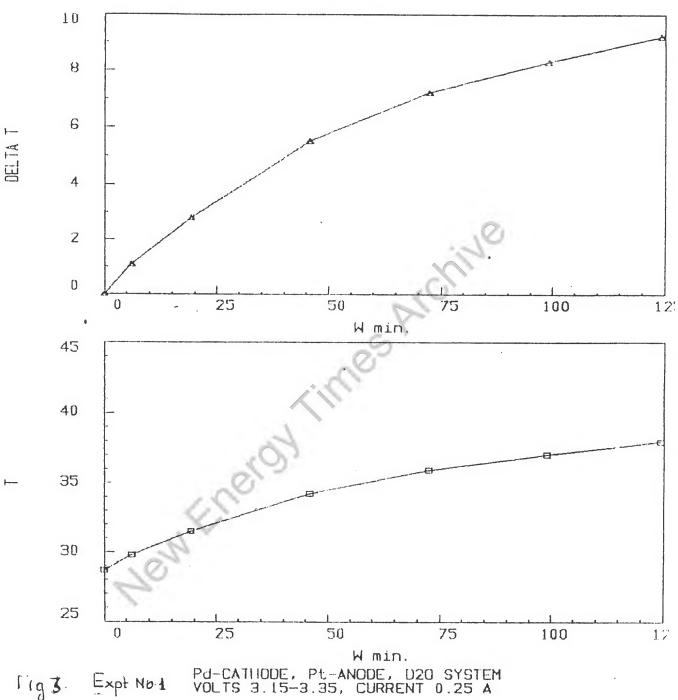
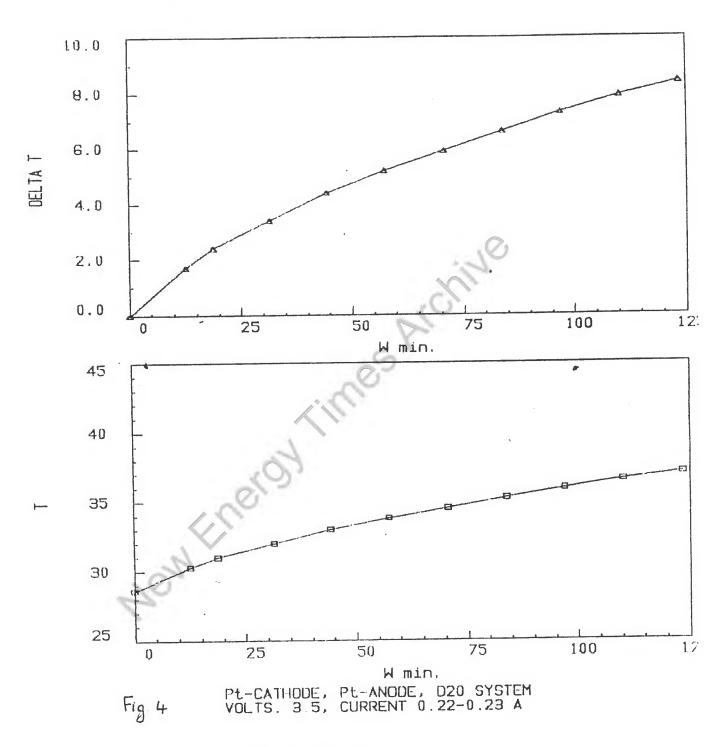


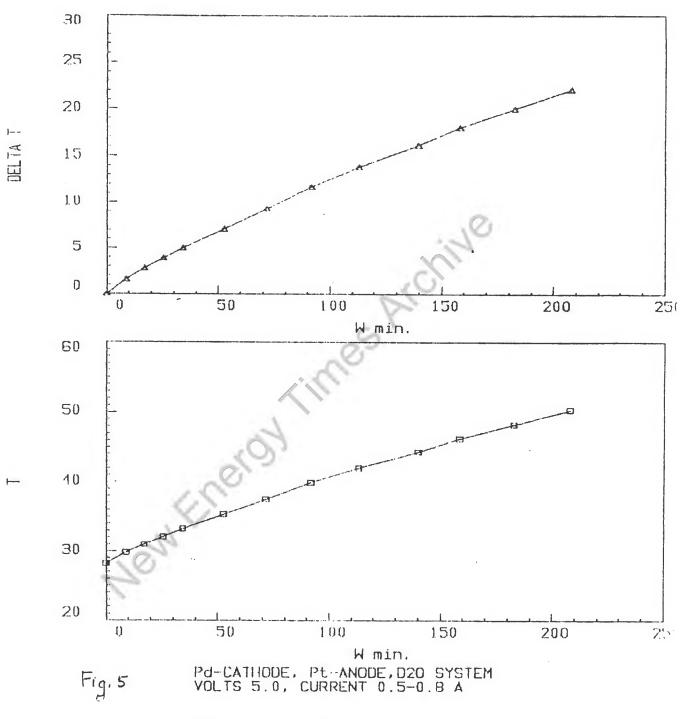
FIG. 2 CURRENT Vs. APPLIED VOLTAGE



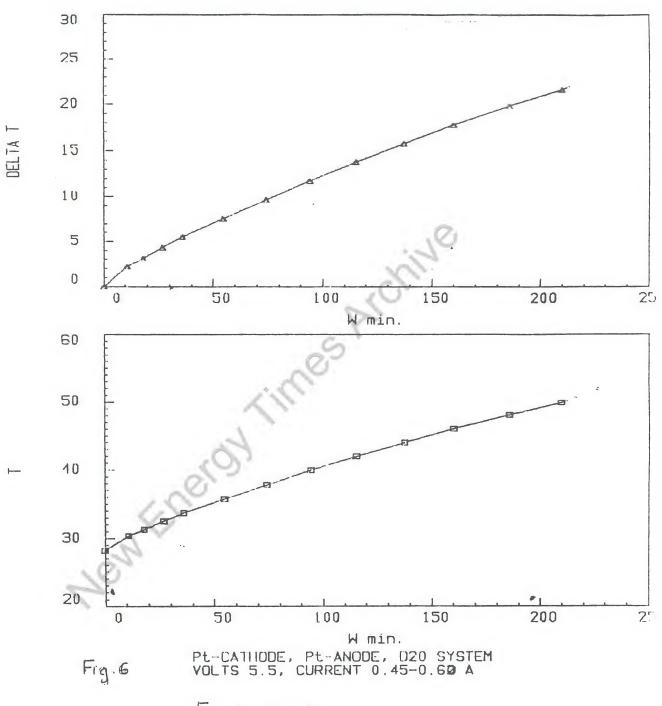
lig3. Expt No 1



Expt : No 2



Expt. No. 3



Expt. No. 4

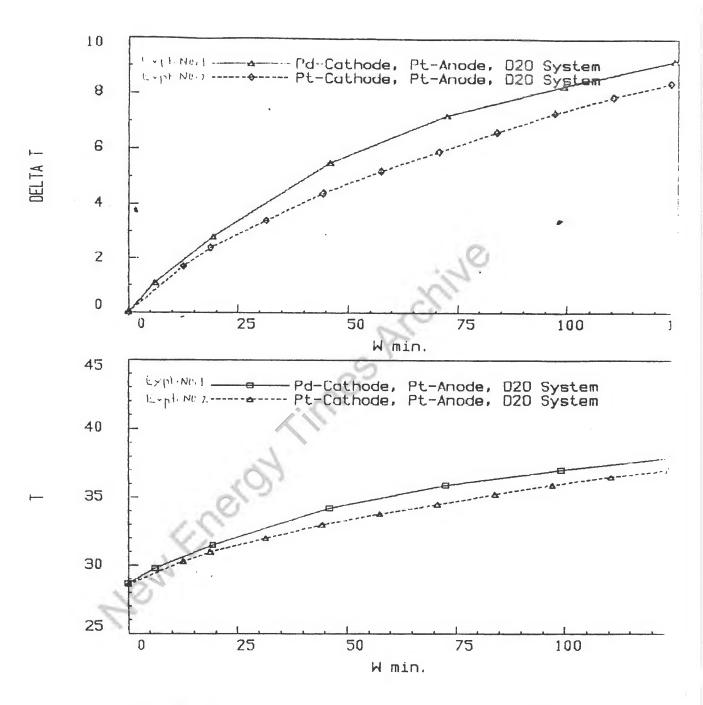


Fig 7. Data from Expls No 1 & 2 are superimposed,

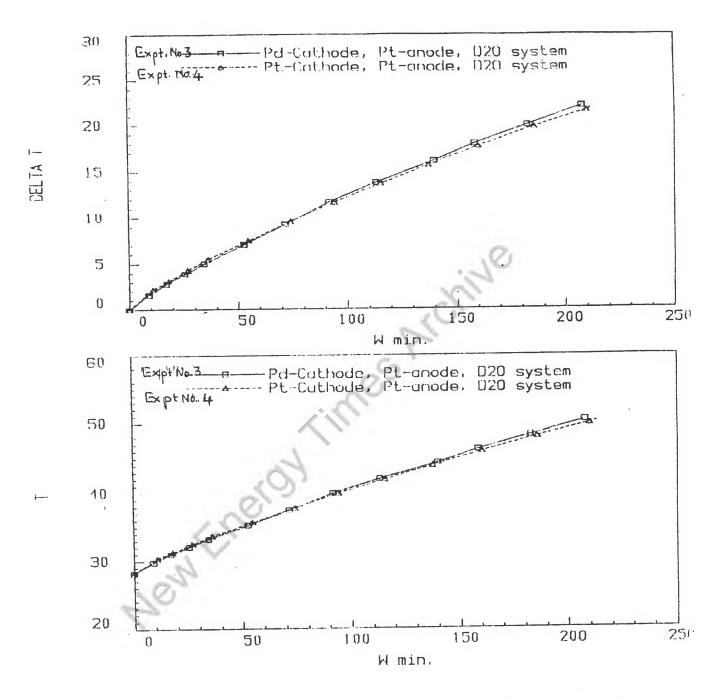


Fig. 8. Data from Expts No 3 and No4 are superimposed.

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The heavy water from the electrolytic cell was sent for tritium analysis elsewhere. It has been reported by oral communication that it contained tritium higher than the blank.

Conclusions:

Excess heating reported by Pons & Fleischmann in the electrolysis experiments of ;heavy water with palladium cathode was not observed in our experiments which were carried out in almost similar conditions. Instead of direct calorimetric measurements, the Pd-D₂O system was compared with Pt-D₂O system. It may be noted that the experiments were carried out for a maximum duration of 285 minutes during which temperature equilibrium conditions were not attained. Effects of deuterium loading on palladium if any, leading to cold fusion as a function of time, was not considered in these experiments.

Further work on cold fusion is in progress in a more systematic way with improved experimental conditions. A large size palladium (1 x 1 x 1 cm cube) cathode, a neutron counter, spacing of electrolyte solution with a suitable gas etc. have been employed in the new experiment. Results of this experiment will be reported soon.

Part II: Neutron Burst Measurements (16th to 22nd June 1989)

Introduction

The initial experiments, the results of which are reported elsewhere, were carried out with a small palladium tube and using lithium hydroxide with heavy water as the electrolyte. There was no indication of any excess heating in these experiments when compared with similar experiments using platinum as cathode in place of palladium. The present experiment was planned with improved experimental conditions for measurement of excess heat and emission of neutrons. Two major neutron bursts were observed. There was some evidence of excess heat in the cell from heat—transfer calculations based on the experimental data. Tritium measurements of the electrolyte were also made by taking samples from the cell during electrolysis. The results obtained in this experiment are discussed in this report.

Experimental

Palladium cathode: A palladium cube of dimensions 1 x 1 x 1 cm was procured from local suppliers. It was heated under vacuum to expel moisture and occluded gases. Electrochemical cleaning of the palladium cabe was carried out by making palladium as anode with respect to platinum cathode in an electrolyte solution of LiOD in D₂O. A current of 50 mA at 1.8V was passed through the solution for about 30 minutes.

Anode: It consisted of a split cylindrical platinum wire gauze surrounding the cathode.

Electrolyte solution: 0.1 M LiOD in D₂D. This was obtained by dissolving lithium metal in heavy water, having an isotopic purity of >99.86% w/w D₂O.

Weight of the electrolyte solution in the cell was 166 g.

Voltage supply to the cell: Electrolysis was carried out at constant current and variable voltage.

Temperature measurements: Cathode temperature was monitored by an MI thermocouple inserted in a cavity made on palladium surface. Temperature of the solution was measured using Pt-100 RTD placed in the solution. Both the temperatures were continuously recorded during electrolysis.

A mechanical stirrer operated by a mini DC motor was used whenever necessary for a thorough mixing of the electrolyte solution.

Gas aparging of the electrolyte solution was continuously carried out initially with nitrogen and later with helium and finally with deuterium gas.

Neutron Detector: A BF3 neutron detector surrounded by paraffin wax was placed below the electrolysis cell. Its counting efficiency was 5 x 10-3 cps/fast neutron as obtained from a calibration using a standard neutron source at the location of the palladium cathode of the experimental set up. A pulse counting channel was connected to the neutron detector. The channel provided integrated neutron count display for a set time (selected for 300) seconds in this experiment) and counting rate signal for recording.

A resistance wire heater was placed in the cell to provide data on electrical heating without electrolysis. This data would be useful for comparison with that from Joule heating during electrolysis.

Photographs of the experimental assembly and the associated instrumentation are shown in Fig.1 and Fig.2 respectively. Schematic diagrams of the electrolysis cell and the associated instrumentation are given in Fig.3 and Fig.4 respectively.

Electrolysis was carried out initially at 600 mA corresponding to a voltage of 5.33 V which gradually dropped to 4.34 V after about 29 hours. The current was then raised to 700 mA for about 41 hours and the to 800 mA and the experiment was continued for a total period of 150 hours (which corresponded to 107.5 Ampere hours). Initially cylinder N2 was passed through the solution. This was changed to helium to eliminate oxygen present as an impurity in cylinder N2. Subsequently D2 was chosen for sparking since it would enhance the deuterium absorption in the palladium cathode. The composition of the cover gas in the cell was also determined occasionally to observe the D2 content. Fresh heavy water was added to the cell from time to time (~53 ml total) to make up the losses caused by electrolysis, sampling and evaporation. Important observations made on neutron emission during the experiment and a summary of the experimental conditions such as gas used for purging, composition of cover gas, cell parameters, etc are given in a chronological order in Table 1.

The electrolyte solution developed colouration towards the end of the electrolysis. This was found to be due to corrosion of stainless steel surfaces of stirrer and RTD. This needs to be avoided in future experiments.

Discussion

Neutron Emission: During the progress of electrolysis, the BF3 neutron counter indicated several neutron bursts as given in Table 1. Fig.5 and Fig.6 show two major neutron spikes observed on the first day of electrolysis, each lasting for about 45 min. and 60 min. respectively. These neutron signals corresponded to about 1 x 10^6 and 2.6 x 10^6 neutrons respectively. Besides these two spikes, there were five more. Since two of these lasted for very short duration, it is not clear whether they were genuine.

Cell Temperature and Excess Heating: The temperature of the electrolytic cell increased during electrolysis and stabilised as shown in Fig.7. The cathode temperature was a degree or two higher that that of the electrolyte solution throughout the experiment. The observed cell temperature data was used to assess the heating characteristics of the cell using a simplified heat-transfer analysis. This is described in more detail in the Annexure. This analysis was carried out for two cases corresponding to two conditions of the experiments as follows

Case A: 4.5 V; 600 ma; stabilized temperature 50.6° C.

Case B: 4.8 V; 800ma; 65.0° C

Table 1

Observations on Neutron Emission and Summary of Experimental conditions in a chronological order

Date	Time (Hours)	CPS (Recorded by BF ₃ Counter)	Purge Gas	D ₂ a in co	over gas	Cell Temp. (° C)	Remarks
15.06.89	0845 - 1730	0.05	-		-		Cirus shut down
	1730	0.15	-		- C)	Cirus 40 MW
16.06.89	1055	0.15	N ₂			28.0	Electrolysis started; Current increased in steps.
	1205	0.15	N ₂	1	-	28.0	Cell voltage 5.33 V; Current 600 mA
•	1245 - 1330	6.0	N ₂		-	34.0- 38.8	Neutron burst (1)
	1415	0.15	N ₂	O_2	10.4 26		
	1452 - 1457	13	N ₂		-		Sharp rise in Cl'S and cell temperature. (45°C →55°C);
	1620	0.15	N_2	D ₂ O ₂	9.0 23.0	48	Neutron burst ?
40	1725 - 1825	18	N ₂				Neutron burst (2). Cathode temp. 52°C; Cell temp. 49.2°C
	1740	2	N_2	D ₂ O ₂	0.1 20.0		
17.0689	1816 - 0940	0.15 0.15	N_2			49.5 53.0	
	0940 - 1030	1	N_2			53.0	Neutron burst (3)
	1207 - 1220	0.6	N ₂ .	D ₂ O ₂	35.0 21.0	53.2	Neutron burst (4)

. 4

<u>Date</u>	Time (Hours)	CPS (Recorded by BF ₃ Counter	Purge Gas	D_ and O_ in cover gas % v/v	Cell Tem (°	
17.06.89	1230	0.15	N ₂			
	1255 - 1331	0.05	N ₂			Power failure; inter- ruption in electro- lysis; Cirus shut down.
	1331 - 0415	0.15	N ₂			Electrolysis started; Cirus at 40 MW.
	1630	0.15	N ₂		48.1	Cell voltage 4.6; Current 700 mA
18.06.89	0415 - 0450	0.8	N ₂	Dicy	54.0	Neutron burst (5).
	0700	0.15	N ₂	5		D ₂ O added to the cell (20 ml)
	0930	0.15	He		55.5	
	1742	5	Не		57.0	Sharp rise in CPS and cell temp. (57°C → 100°C; Neutron burst ?
19.06.89	0255 - 0930	1.2 (Steady)	Не	D ₂ 55.9 O ₂ 25.3	56.0 56.0	Interference from Cirus (Tray rod work)
	0930	0.15	Не		56.0	Cell voltage 4.89; Current 800 mA
7	1020	0.15	Не			Sample (6.2 ml); Electrolyte added (6.2 ml); D ₂ O added (7.9 ml)

Date	Time (Hours)	CPS (Recorded by BF ₃ - Count er	Purge Gas		and O	Cel.	
20.06.89	1200	0.1	Не	D ₂ O ₂	30 14	59.9	Cell voltage droppe by itself to 4.4 V; Current 630 mA
	1348	0.1	Не				Current raised to 800 mA; Cell voltage 4.9 V
	1825	0.2	Не		10		D ₂ O added (20 ml); Cell temp. (62°C → 59°C)
21.06.89	1000	0.05	He	D ₂ O ₂	7	65.0	Cirus shut down. Sample (7.5 ml); Electrolyte added (7.5 ml)
	1120	0.05	He				D ₂ O added (11.5 ml)
	1452	0.05	D ₂			62.0	2
22.06.89	0945	0.05	D ₂	D ₂ O ₂	68 30	68.0	
	1515	0.05	D ₂				Cell current was reduced in steps
	1620	0.05	D ₂			60.0	Electrolysis terminated.

---000000000---



FIG. 1 - EXPERIMENTAL ASSEMBLY
FOR ELECTROLYSIS

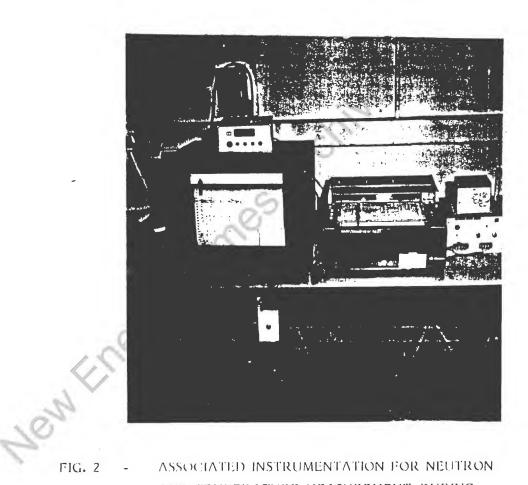


FIG. 2 - ASSOCIATED INSTRUMENTATION FOR NEUTRON
AND TEMPERATURE MEASUREMENT DURING
ELECTROLYSIS

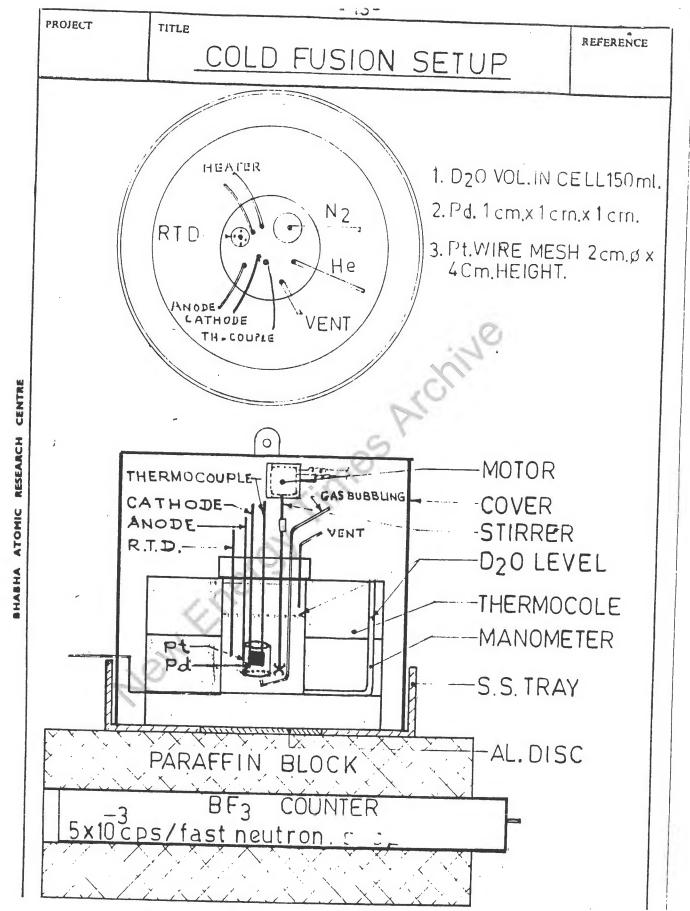
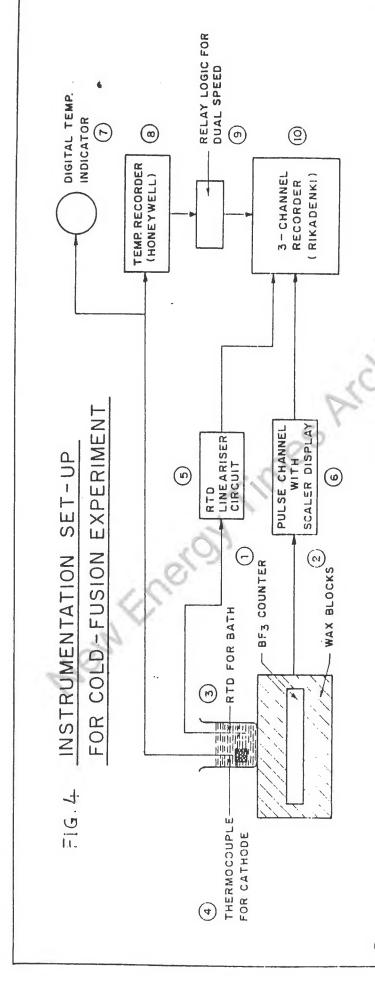


FIG. 3. CELL FOR ELECTROLYSIS



- (1) BF3 COUNTER FOR NEUTRON COUNTING HAVING SENSITIVITY OF ABOUT 60 COUNTS PER Sec./ NEUTRON FLUX.
- (2) WAX BLOCKS FOR THERMALISING NEUTRONS.
- (3) Pt-100, 3 WIRE R.T.D. FOR MEASURING ELECTROLYTIC BATH TEMPERATURE.
- (4) MINERAL INSULATED, UNGROUNDED TYPE-T THERMOCOUPLE IN 3.2 mm. DIA. S S SHEATH, TIP EMBEDDED IN PALLIBIUM CATHODE.
- (5) LINEARISER BRIDGE CIRCUIT, CONNECTED TO R.I.D., TO CONVERT C-100 °C TO 0-100 mv OUTPUT.

- (6) PULSE COUNTING CHANNEL HAVING SCALER/TIMER DISPLAY FOR NEUTRON COUNTS AND 0-10 V OUTPUT FOR 0-1 TO 10 \$ cps.
- (7) DIGITAL TEMP.INDICATOR, CONNECTED TO THERMOCOUPLE.
- (B) HONEYWELL TEMPERATURE RECORDER, FOR CATHODE TEMPERATURE (0 TO 400 °C)
- (FOR 10 MINUTES) WHEN CATHODE TEMP. EXCEED\$125 °C
- (10) 3 PEN-"RIKADENKI" RECORDER TO RECORD
- i) NEUTRON COUNTS FROM 0.01 cps TO 104 cps AND ii) BATH TEMP. FROM 0 TO 100 °C

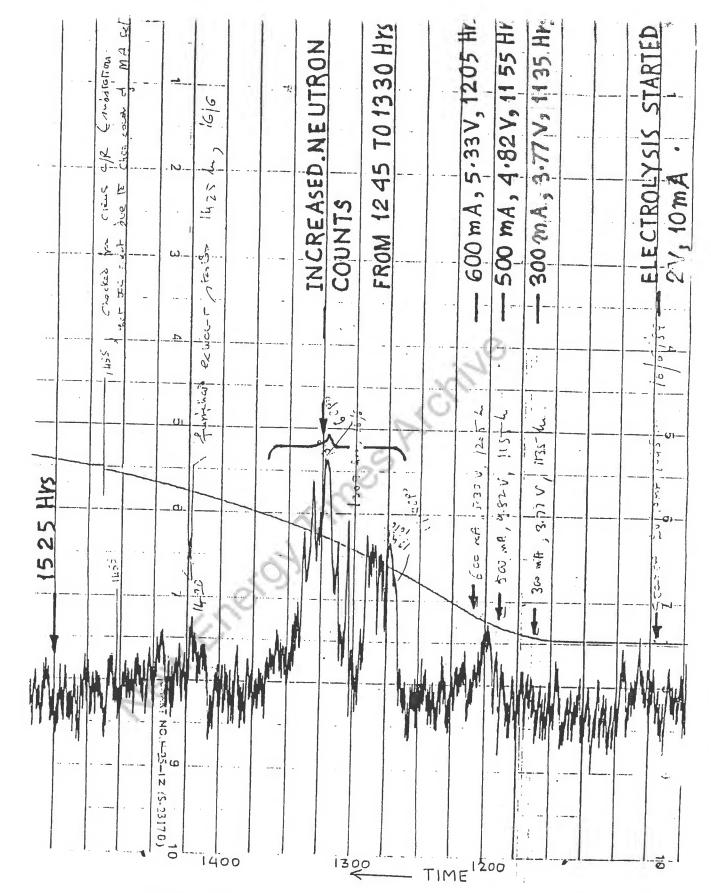


FIG. 5. NEUTRON SPIKES VS TIME

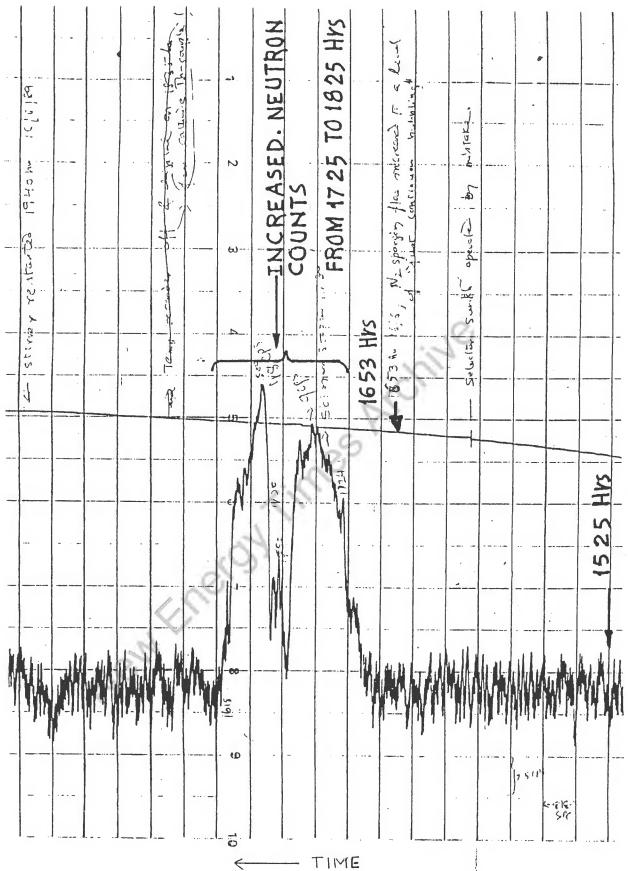


FIG.6. NEUTRON SPIKES VSTIME

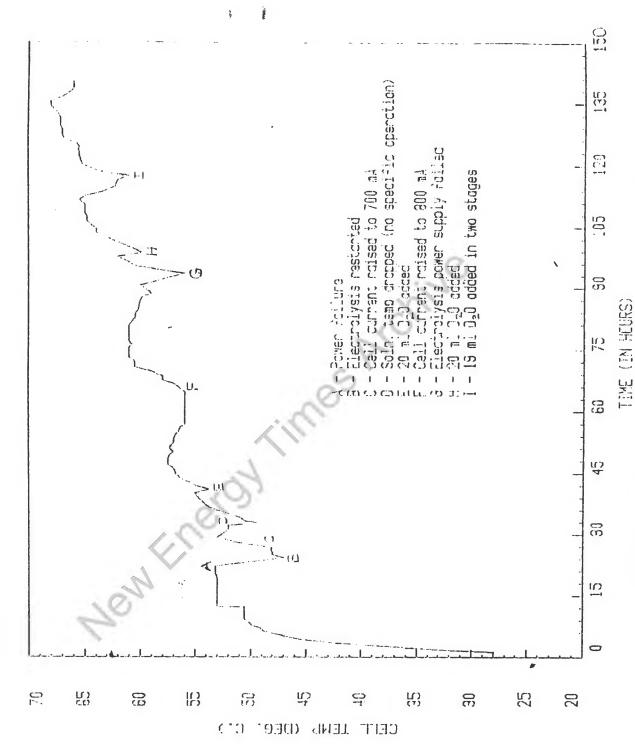


FIG. 7. - CELL TEMPERATURE VS TIME OF ELECTROLYSIS.

AC=30.005

0

RCNG cold fusion expension No I with Pa IXIX Icus in return 1, CIRL'S	16 6.84 15 22.6.89 Angere- hours 107.5 Pa unloudeing commenced of 1515 hrs 17:55 22 JUN 89	2 9 4 L	400 000 00 00 00 00 00 00 00 00 00 00 00		NOS.		
RCNG		(() = 10000	10.000 KIIV VIIW				

() () () C=15.005 KEV INT = 844 Went Flordy Lines Williams Went Flord August 1988

The thermal time constant of the system was determined from the experimental data. Using the time constant, water equivalent of the cell, and the observed stabilized temperature rise (with respect to ambient), the heating rate of the cell was computed. This was found to be 2.47 W for Case A. Based on the heat transfer analysis and observed temperature rise in Case A, the expected stabilized cell temperature rise in Case B was computed to be 29.70 C. However, the observed temperature rise in Case B was 370 C. Heating rate estimated from the experimental data in this case was 4.05 W.

It can be seen from the calculations in the Annexure that the Joule heating for Case A and Case B are 1.5 W and 2.28 W respectively. Thus, the results obtained from the heat—transfer analysis seem to indicate excess heating of 0.97 W for Case A and 1.77 W for Case B. However, these excess heating values are smaller than the estimated lower limit values of 3.8 W and 5.2 W for Case A and Case B respectively interpolated from the excess heating data on cold fusion reported by Pons and Fleishmann in their paper (J. Electroanal. Chem. 261, p 301, 1989).

The palladium cube, after 150 hours of electrolysis, was counted for 21 KeV Pd-K-shell X-rays that might have been excited by protons produced via the nuclear reaction D + D — T + p. A chart recording for this counting is given Fig.8. There was no positive indication for the Pd-K-shell X-ray emission.

The cell heavy water samples were sent for tritium analysis. The results will be reported elsewhere.

Conclusions:

The present experiment has given evident for neutron emission and possible excess heating arising from reported cold fusion. Tritium results would throw further light on the observed phenomenon.

During electrolysis, the liberated gases D₂ and O₂ may recombine in presence of palladium (acting as a catalyst) producing heat. It is assumed that palladium which was inside the electrolyte solution would not catalyse such a reaction. Also, above the solution, such a reaction is believed to be not significant due to absence of a suitable catalyst.

During degassing of the palladium cube on heating under vacuum for use in the next experiment, it was observed that a lot of accumulated gases continued to evolve for almost an hour.

Experiments are being planned to collect additional data on the subject.

Part III: Analysis of Observed Cell Temperature Variations

Introduction

In a typical run of the experiment (Case—A) conducted on June 16, 1989, a cell voltage of ~4.5 volts and a constant cell current of ~600 mA were used and the neutron count rates and the electrolyte as well as the palladium cathode temperatures were monitored periodically. The stirrer was intermittently operated during these measurements.

Subsequently another experiment (Case-B) was carried out with a call voltage of ~4.85 volts and a constant cell current of ~800 mA.

Table—1 summarises the temperature variations observed till the system reached equilibrium.

Since it is complex to arrive at the heat transfer conditions of the system from first principles, the observed time variation of the cell temperatures in the Case—A experiment (4.5V, 600 mA) were used to obtain the thermal time constant o the system. Using the known water equivalent value (W) of the major contents of the cell, the effective product of the heat transfer coefficient to the ambient heat sink and the heat transfer area of the system (hH) eff for this experiment was estimated. Finally the cell heating rate (Pcell) is obtained as the product of (hA) eff and the observed stabilised temperature rise of the cell with respect to the ambient temperature. This 'observed' cell heating rate was compared with the expected cell joule heating for the voltage and current values used for the experiment.

Based on the above approach, an assessment was also done for the expected stabilised cell temperature for case—B experiment (4.85 V, 800 mA) for comparison with the observed value.

Finally the expected cell temperature variations with time were assessed for the heat transfer conditions of the experiment, but assuming ;that the cell heating is due to a) only the joule heating.

b) joule heating plus an estimated lower value of the cathode heating due to cold fusion

c) joule heating plus an estimated upper value of the cathode heating due to cold fusion events.

The cathode heating rates were estimated using an approximate formula obtained by fitting the experimental data given by Stanley Pons and Martin Fleishmann.

Cell Heat Transfer Analysis

For a system with a heating source P, water equivalent W, heat transfer area A, effective heat transfer coefficient to ambient heat sink h, and ambient sink temperature Ts, the volume average system temperature T, will be given by

$$W \frac{dT}{dt} = P - h.A. (T - T_g)$$
 (1)

With the assumption that initially the cell and the heat sink temperatures are different and that the heat sink temperature does not vary with time, the solution of equation (1) with the scale of $T_{\rm g}=0$ is given by

$$\Delta T(t) = \Delta T (0)e^{-t/\tau} + \Delta T (\omega) (1-e^{-t/\tau})$$
 (2)

where $\Delta T(t) = P/(hA)$ and $\Delta T(t)$ is the excess of cell temperature at time 't' over the sink temperature T_s and

$$\tau = W/h\Lambda \tag{3}$$

where au is the effective system thermal time constant.

Assuming that initially the cell and heat sink temperatures are equal, the cell thermal time constant can be approximately estimated from equation (2) using the

derived relation

$$\tau = t/Log_{e} \left\{ \frac{\Delta T(\omega)}{\Delta T(\omega) - \Delta T(t)} \right\} \qquad (4)$$

The observations for the Case—A experiment given in Table—1 were used to estimate the thermal time constant. The mean value from different data works out to be about 2.04 hour, for the cold fusion experimental set up in the Reactor Chemistry Section Laboratory (Room No. 41, Cirus).

It may be noted that theoretically the stabilised cell temperature (excess over heat sink temperature) is directly proportional to the cell heating rate Pcell (for given set up and heat transfer conditions).

Thus the assessed cell heating 'Pcell' for the Case—A experiment can be worked out using the observed stabilised cell temperature rise using

Pcell =
$$\Delta T (\omega) x (hA)$$
 eff
and (hA) eff = W / τ (5)

Note: This calculational model has been verified to yield satisfactory results (within about 10%) when typical cell contents were heated by electrical resistance heaters with known heating. For the details of this calibration testing, please see Appendix—I attached.

For the cell used for the experiments, the estimated water equivalent is W = 191.4 cal / degC as given below:

Major component (Cal/degC)	Mass (g)	Sp.ht	Water Eq.
Glass Beaker	125	0.1988	24.85
Heavy Water	165	1.0	165.0
1cm cube Pd	11.4	0.061	0.695
Pt Anode	26	0.33	0.858
		Total	191.4

Thus (hA) eff = 191.4/2.04 = 93.8 Cal/hr/degC

Since ΔT (∞) observed is 22.6 degC for the (4.5V, 600mA) experiment,

Pcell =
$$\Delta T$$
 (ω) x (hA) eff = 22.6 x 93.8 cal/hr = 2120.4 cal/hr = 2.47 watts

The assessed value Pcell for the experiment may be compared with the expected cell joule heating Pj responsible for heating the cell contents (in the absence of any additional heating produced by possible "cold fusion" events in the palladium cathode).

$$Pj = (Vcell - VO) \times Icell = (4.5 - 2) \times 0.6$$

= 1.5 watts

where Vcell is the cell voltage applied, Icell is the cell current and Vo is the thermoneutral potential for the cell (upto which no cell current is possible).

The value of Vo for the cell used was experimentally found to be Vo = 2 volts.

Thus the observed incremental change of system stabilised excess temperature over heat sink temperature for the cell leads to a value of 22.6 / 2.47 degC/Watt = 9.1 degC/Watt.

Comment:

It may be noted that the assessed cell heating (2.47 watts) purely from the (4.5V, 600 mA) cell experimental data is LARGER (by about 0.97 watts) than the expected cell joule heating (1.5 watts). This conclusion is significant. However before attributing this discrepancy to cold fusion events in the palladium cathode, it is worthwhile to eliminate/minimise all unwanted heat inputs to the cell such as that due to the electrical stirrer and to accurately know the cell joule heating and repeat the experiment carefully.

Using the above approach one can also assess the expected cell equilibrium temperature for the Case—B experiment conducted on 20—6—89 with the same experimental set up used for the Case—A experiment. In this case the cell temperature stabilised at 65°C with an ambient of 28°C, leading to a cell temperature rise of 37°C.

However assuming the heat transfer parameters assessed for the Case—A experiment are applicable for Case—B experiment, one would expect a cell temperature rise of

$$(22.6 / 2.47) \times [(4.85 - 2) \times 0.8 + (2.47 - 1.5)] =$$

 $(22.6 / 2.47) \times [2.28 \text{ watts} + 0.97 \text{ watts}] = 29.7 \text{ C}$

to be compared with the observed value of 370 C.

The assessed cell heating using the observed stabilised cell temperature rise works out to be 37°C x 93.8 = 3471 Cal/hr = 4.05 watts. for the Case—B experiment. It may be noted that the additional heating of 0.97 watts observed over the estimated joule heating of 1.5 watts in the Case—A experiment is already corrected for in this assessment.

Comment:

Thus the assessed cell heating (4.05 watts) is also LARGER than the expected joule heating of 2.28 watts in the cell experiment with (4.85 V, 800 mA). Anticipated cell temperature variation with time for the assumed palladium cathode heating due to cold fusion events. To compare the observed cell temperature variations with time with the expected values if the palladium cathode has heating due to cold fusion, calculations were done using equation (2). An approximate value for the cathode heating was obtained using a least square fitted relation as a double conic function of palladium cathode rod diameter and cathode current density.

$$Q = 0.00855 \times D^{0.364} C^{1.13}$$

where Q = Excess heating rate in Pd cathode (W/cc)

D = Pd rod diameter (mm)

and C = Cathode current density (mA/sq.cm)

The palladium cathode excess heating rate data given by Stanley Pons and Martin Fleishmann [in their original paper "Electrochemically induced nuclear fusion of Deuterium", J. Electroanal Chem, 261 (1989)] were used for this fitting. The fit has a standard deviation of 18.3% for the nine data used as given below. For each value of Pd diameter, the first line gives the reference data and the second line gives the fitted value, as a function of the cathode current density.

Pd Rod Dia	Excess Heatin 8 mA/sqcm	g Rate (W/cc) for (64 mA/sqcm	Current density' 512 mA/sqcm	ensityʻ A/sqcm	
1.0	0.095 0.089	1.01 0.9 3	8.33 9.70		
2.0	0.115 0.115	1.57 1.20	9.61 12.5		
4.0	0.122 0.148	1.39 1.54	21.4 16.1		

The equivalent rod diameter of the 1 x 1 x 1 cm cube of palladium used is 11.28 mm. The current density would be in the range of 100-111111150 mA/sqcm for Case-A and in the range of 1333-200 mA/sqcm for Case-B.. Using the fitted relation, the estimated lower limit excess heating in the cathode are 3.76 W/cc and 5.19 W/cc for Case-A and Case-B respectively. The corresponding upper limit values are 5.94 W/cc and 8.23 W/cc for the two cases.

For Case—A experiment, calculations were done using equation (2) for the anticipated cell temperature variation with time for the lower and upper limit values of the excess heating in the cathode in addition to the estimated joule heating of 1.5 Watts. The heat transfer condition was assumed to be characterised by the estimated thermal time constant of 2.04 hour and by the (hA) eff value of 93.8 Cal/h/degC.. The computed upper and lower limit stabilised cell temperatures are 48.1 C and 68.0 C. Figure 11.1 gives the results. For comparison, curves for only joule heating of the cell and the actually observed temperatures (for assessed cell heating of 2.47 watts) are also indicated in Fig—1.1.

Comment:

It is seen that the observed cell temperature variations with time are much lower than those anticipated with estimated excess heating in the cathode. However they are significantly higher compared to the cell condition with only calculated joule heating present. Thus additional experiments with carefully controlled heat transfer conditions and higher values of cathode current density than that used in Case—A would be worthwhile.

Recommendations

Since both the Case-A (4.5 V, 600 mA) and Case-B (4.85 V, 800 mA) cell experiments seem to indicate larger than expected (joule) heating in the cell, it is

worthwhile to repeat the cell experiments carefully with:

(i) unwanted (but accurately known) heat inputs into the cell eliminated/minimised,

(ii) avoiding thermal contacts of the beaker surfaces with surrounding structural metal components (if any) so as to facilitate accurate estimation of cell water equivalent value and

(iii) increased cathode current density values at reduced cell voltages to minimise joule heating for easier resolution of the excess cell heating due to cold fusion events if they occur. Closely spaced cylindrical geometry electrodes and increased electrolyte electrical conductivity values may be helpful in this regard.

Run of (June 16, 1989)

Observations with Resistance Heating of the Cell

Calculations were also done for a simulation cell experiment carried out on 23.6.89, in which the cell contents were heated by electrical resistance heating, aimed to simulate the experimental conditions used for case—A and Case—B experiments. The heat input to the cell was known from the voltage and current values and the actual fraction of the resistance wire immersed in the electrolyte used for resistance heating experiment.

During the resistance heating experiment, stabilised cell temperatures were noted for three different sets of cell voltage and current values, (viz.) (4.5V, 555 mA), (5.7V, 700 mA) and (6.4V, 780 mA).

The observed time variation of the cell temperature for the (4.5 V, 555 mA) was analysed using the "thermal time constant method". The cell heating rates were then assessed for comparison with the heating values, known from current and voltage values used. It may be mentioned that, there was no electrolysis involved in these experiments.

Using the 'time constant' method the cell temperature variations with time for the simulation experiment with resistance heating were analysed. These observations are given in Table-1, and the estimated mean time constant value from different data was 3.62 hour. Thus the heat transfer conditions for the resistance heating experiment are not quite similar to those for case-A (for which a thermal time constant value of 2.04 hour was assessed from its experimental data). This is possibly due to the absence of electrolysis' (and associated gas release phenomena) and due to changes in effective thermal environmental conditions of the cell, which could influence heat losses from the system used for the resistance heating experiment.

Following a similar procedure has for case—A, the cell heating value is assessed to be Pcell = 1.67 watts for the resistance heating experiment, to be compared with the actual heating value of 1.5 W, as given below:

$$(hA)eff = W/\tau = 191.4/3.62 = 52.9 cal/hr/C$$

 $\Delta T() = 27^{0}C \text{ (observed)}$
 $Pcell = \Delta T_{\infty})..() x (hA) eff = 27 x 52.9$
 $= 1428.3 cal/hr = 1.67 Watts$

The actual cell heating value can be computed from the voltage and current values used for resistance heating and is given by

4.47 volts x 555 mA x (36.3/60) = 1.50 watts

where the factor (36.3 cm/60 cm) represents the ratio of the resistance heating wire immersed in the electrolyte to the total length of the resistance wire used in the experiment. The additional correction factor due to a small length of the wire within the cell above the electrolyte level was experimentally found to be negligible.

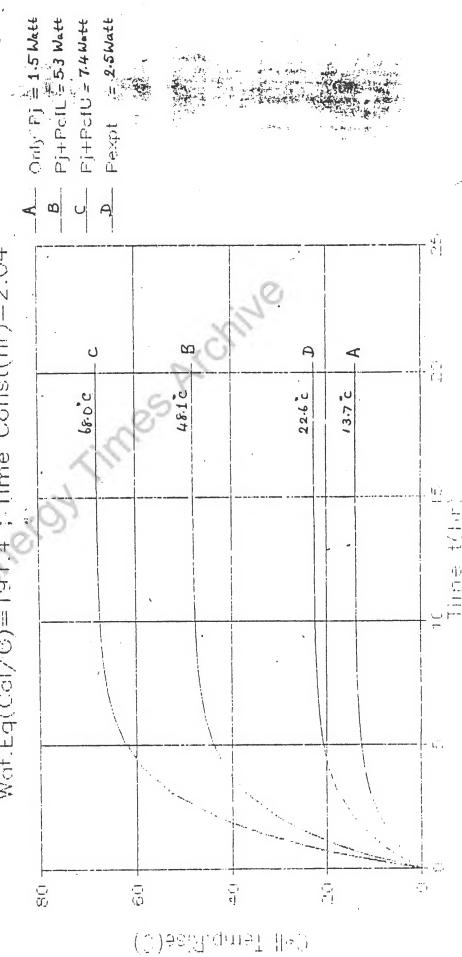
Similarly the cell resistance heating values for the (5.7V, 700mA) and (6.4V, 780 mA) portions of the experiment were also assessed using estimated time—constant and the observed stabilised cell temperatures. These are tabulated below:

Voltage(V)	Current(mA)	Temperature	Cell Heat.	Time Const
4.5	555	27.0	1.67	1.50
5.7	700	40.5	2.50	2.41
6.4	780	46.1	2.85	3.02

The good agreement (within 10%) between the assessed cell heating value (using the thermal time constant method) and the actual resistance heating gives confidence for the application of the thermal time constant method for the cell experiments.

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Cold Fusion Expt(Vcell=4.5V;Icell=600 mA) wat.Eq(Cal/C)=191.4 ; Time Const(hr)=2.04



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Table 1. Summary of Observations during Electrolysis

	(DegC 1.5 1.5 42 47 45	28 C 2.8 14.8 5.4 17.4) O 3
1415 4.79 600 1505 4.66 600 1600 4.58 600 1700 4.53 600	47 45	2.8 14.8 5.4 17.4	
1900 4.48 600 2000 4.5 600 2200 4.45 600 2300 4.42 600	52 49 52 50 52 50	7.2 19.2 8.8 20.8 9.2 21.2 0.1 22.1 0.6 22.6 0.6 22.6	3 2 L

Thermal Time t Temp Rise [T(inf) - Ts] [T(inf)-T(t)] Time Cons (hour) (DegC) (hour) 1200 0 1415 2.25 14.8 17.4 2.897436 2.115007 1505 3 4.346154 2.041801 1600 4 19.2 6.647059 2.111738 1700 5 20.8 12.55556 1.976157 1755 21.2 16.14286 2.157127 1900 22.1 45.2 1.836741 2000 22.6 1-836741 2200 Av.Time Const = 10 22.6 2.039762 2300 11 22.6

Table-1: Cold Fusion Experiments in ROMG(June 23, 1989) (With 1 x 1 x 1 cm Palladium Cathode and 2 cm dia x 4 cm long Platinum Anode) Resistance Heating Run

Experiment	No.	#1	Date:	23-6-89	Ts(DegC)=	26 5
		11	2400.	45 0 05	rs (nedr) =	1.0

Time	Voltage (V)	Current (mA)	Solution RTD	Temp(C) Th.Coup	Temp Ris (DegC)	Time Int t(hour)
1100 1115 1190 1230 1330 1430 1600 1800 2000 2200 0 200 400 500 600	0 4.48 4.47 4.46 4.46 4.47 4.47 4.47 4.47 4.47	0 0 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	26.5 26.5 26.5 33 37.8 41.5 45.6 49 51.5 52 53 53.5 53.5	27 27 27 34 38 42 46 50 52 53 53	0 0 0 6.5 11.3 15 19.1 22.5 25.5 25.5 26.5 27 27	0 1 2 3 4.5 6.5 8.5 10.5 12.5 14.5 16.5 17.5 18.5
700	4.47	555	53.5	53	27	19.5

		^			
Time Int t (hour)	Y =	[T(inf)-	rs] r(t)]	Time Const - (hour)	- t
-		4 -		-	,
. 0	20	9° I		-	
1	00	1.317073		3.630924	
3		1.719745		3.688838	
4.5		3.417722		3.661591	,
8.5		13.5		3.661591 3.661591	
10.5 12.5		18		3.661591	
14.5		18 54		3.627719 3.265852	
16.5 17.5		lean Time	Const	3.617683	
18.5	•	reas IIME	COMBC	3.01/663	
19.5					

Search for Nuclear Fusion In Gas Phase Deuteriding of Titanium Metal

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The possibility of D-D nuclear fusion in some deuterium-metal systems, under ambient conditions, has aroused feverish world wide interest. Most of the work reported, so far, concerns deuterium charging of Pd metal through electrolysis of D₂0.

In Chemistry Division, we have carried out some experiments on the deuteriding behaviour of Ti metal, through gaseous route, in the absorption as well as desorption modes, with the view to look for the fusion products, neutrons in the present case. This kind of experiments have been reported by Frascatti Group in Italy. These authors detected neutron emission lasting over a period of several hours.

Experimental

Experimental arrangement for deuteriding Ti metal is shown in Fig.1, which is self—explanatory. This set up has been routinely used for high pressure hydriding studies on several systems, reported by us (1-3). In some of the experiments reported here, deuterium pressure was cycled between high and low values by simply changing the temperature of the cell housing the sample. Most of the experiments were done in the desorption mode. Ti metal pieces (cut from a sheet) were surface cleaned and subjected to activation treatment before D_2 loading and subsequent desorption treatment etc.

Neutron counting set up consists of an array of 24 He³ counters arranged in a well like geometry. These counters diameter (each 50 cm in length 2.5 cm in diameter and filled with He³ at 4 atm.) housed in paraffin moderators, are all connected in parallel to a single pre—amplifier. The counting efficiency of this system was found to be 10%. The counts are recorded in 8192 channel multi—scalers. In the experiments reported here dwell time of 40 sec. was fixed, so that each point in Fig.(2) to (5) represents the number 4* of counts per 40 sec. The back—ground counts collected for about 10 days, before the start of these deuteriding experiments, was found to be quite steady 60 counts/40 secs. This back—ground count rate continues to be the same well after our experiments.

Results:

In the first set of experiments, starting from 3rd June 1989, after activating Ti metal pieces, D2 Qas was contacted with the sample at a pressure 10 atm. while keeping the sample at low temperature (~77K). After a soaking time of ~20 min., sample temperature was raised gradually, while simultaneous evacuation was started. Within about 15 min. the neutron counter registered an increase in count rate reaching a max. 3900 (as compared to back—ground counts of ~60), see Fig.2. On withdrawing the reactor from the counting well, a considerable reduction in the counts was observed. On re—introducing the reactor after background counts are restored, an additional peak like structure was observed. Although the evacuation was continued, no further increase in count rate over the background could be observed over the next twenty hours.

Next experiment on the same charge was carried out by repeating the conditions of first experiment. The results of this experiment, dated 4th June 1989, are shown in Fig.3. Again two peak like structures, each lasting for about 30 min. and separated by 50 min. were seen. However, the intensities of both these structures are greatly reduced, as compared to the first experiment (3rd June 1989), the max. Counts being ~.700)

In the third experiment, with the same charge of Ti pieces, D2 gas pressure was made to cycle between ~50 atm. to 13 atm. by changing the reactor temperature from room temperature to 77 K. In this case large changes in counts, temperature to 77 K. In this case large changes in counts, as a function of time were noticed. An increasing trend of counts initiated at ~2330 hrs on 4th 3 June 1989 lasted for almost 7 hrs. with an estimated integral counts ~6.5x105. Even after this long bursts like structures, some additional peaks were observed on 5th June 1989. With no further structures observed over the next few hours, desorption was carried out after loading the sample with D2 has with the sample temperature at ~77K. By raising sample temperature gradually, while simultaneously evacuating, a much bigger structure lasting for ~2 hrs. (from ~1830 to 2030 hrs. on 7th June 1989) Was seen (Fig.4). An approximate estimate of integrated count over this period is $7x10^5$ Further experiments with this charge, involving D2 loading followed by prolonged periods of evacuation at temperature upto a max. of ~200°C, did not show further structures.

Second series of similar experiments on a fresh charge of Ti from the same source did not exhibit exactly similar behaviour, as found for the first charge. However, one set of experiments on 17th June 1989 (see Fig.5) involving pressure cycling, followed by evacuation, exhibited increase in count rate lasting over a period of ~100 min. In this case, the scatter in the counts was found to be rather large and maximum counts upto $10^5/40$ sec. were observed, as compared to back—ground count of ~60/40 sec. This charge showed no further increase in count rate even after various treatments.

Further experiments are planned — (i) to study all possible parameters relating to the observed increase in the count rates, (ii) to identify the source of these extra counts, and (iii) to investigate the energy and time structure of the radiation responsible for the observed peak like structures.

Acknowledgement

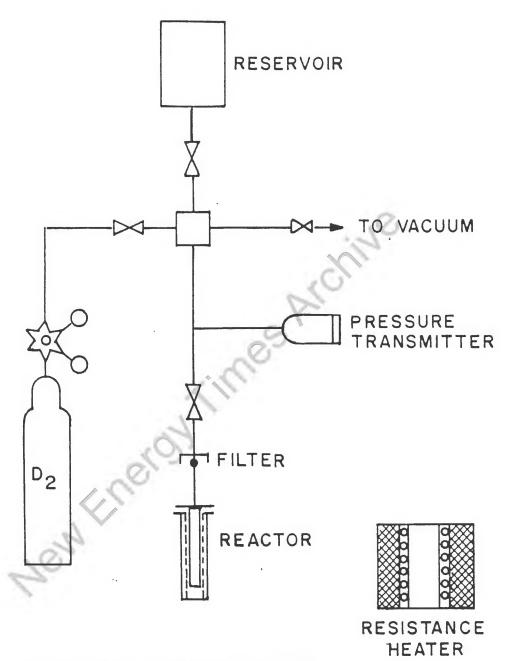
Authors are thankful to Dr R.M. Iyer, Director, Chemical Group for encouragement and helpful discussions. Thanks are also due to Dr P.R. Natarajan, Head, Radiochemistry Division and his colleagues for providing neutron counting facility.

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SCHEMATIC DIAGRAM OF THE HYDRIDING / DEHYDRIDING UNIT

FIG. 1

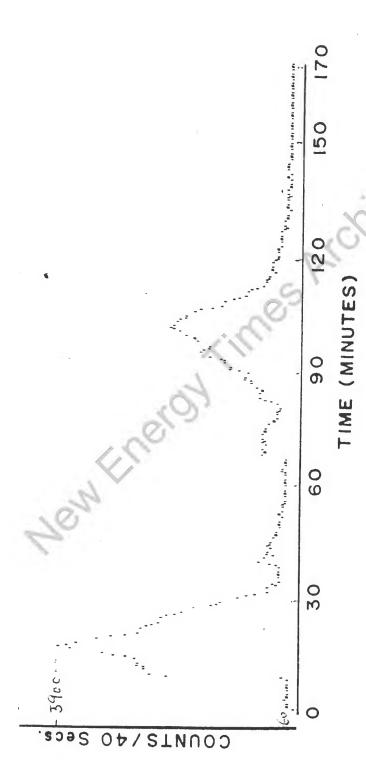


Fig. 2 Desorption mode experiments (3rd June 1989)

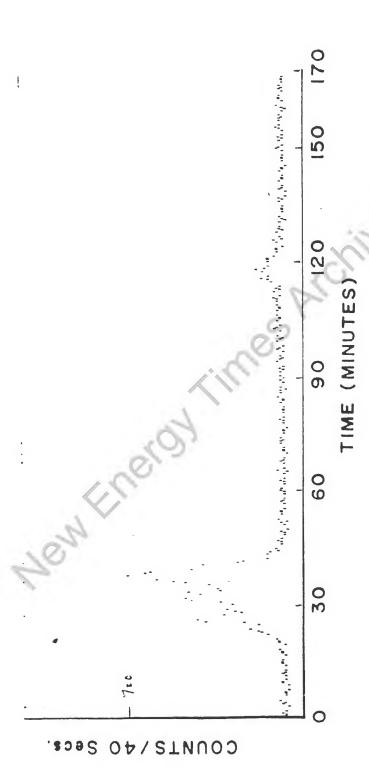
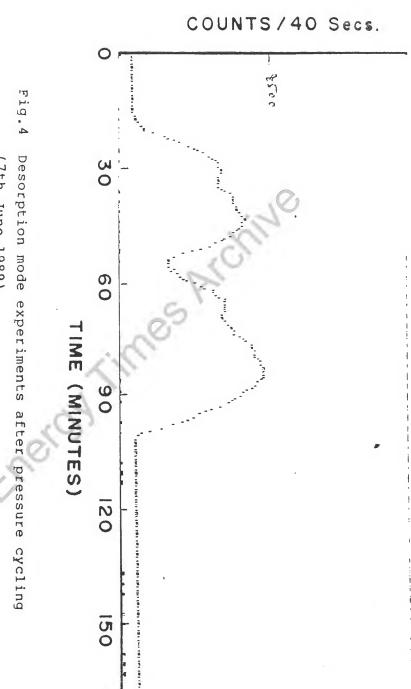
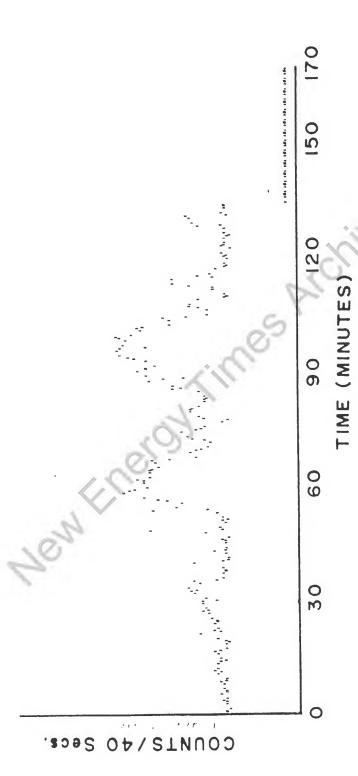


Fig. 3 Desorption mode experiments (4th June 1989)



(7th June 1989)



Desorption mode experiments after pressure cycling with second charge of Ti (17th June 1989) Fig.5

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Deuteration of Machined Titanium Targets for Cold Fusion Experiments

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Introduction:

(fold fusion experiments were conducted in solid targets made from titanium loaded with deuterium gas. The absorption of deuterium by Ti is a reversible process and when titanium is heated in a deuterium atmosphere, the reaction will continue until the concentration of deuterium in the metal attains an equilibrium value. This equilibrium value depends on the specimen temperature and the pressure of the surrounding deuterium atmosphere. Any imposed temperature or pressure change causes rejection or evolution of deuterium until new equilibrium state is achieved. If the surface of titanium is clean, the rate of absorption increases rapidly with temperature. At temperatures above 500 C, the equilibrium is achieved in a matter of few seconds and so deuteriation of targets can be achieved in a few seconds time. However, deuterium absorption is considerably reduced if the surface of Ti is contaminated with oxygen. Keeping in view these facts, a procedure was evolved for titanium target preparation and subsequent deuterium absorption. The following sections describe the details of preparation of the targets, chemical cleaning of the targets and degassing and deuteriation of the targets.

Preparation of the Targets:

Titanium targets of different sizes and shapes (planer, conical etc) were prepared. Targets were typically fraction of a gram in mass and were machined out of a Ti rod using Tungsten carbide tools with continuous cooling arrangement. Care was taken to avoid overheating during machining because any overheating hardens titanium and could spoil it.

Chemical Cleaning of the Targets:

Machined pieces were first degreased with acetone and then in an ultrasonic bath. Then the oxide layer was removed by immersing the sample in a 1:1:1 mixture of water, nitric acid and sulfuric acid. The targets were rinsed in water and acetone and then dried. HCl treatment was given to form an adherent hydride layer on the surface. Targets thus prepared can be preserved in a moisture free environment prior to deuterium absorption.

Degassing and Deuteriation of Targets:

Targets were first degassed by heating to ~900 C in a glass vacuum chamber using a 3kW, 2 MHz induction heater. Degassing was continued till a vacuum of less than 2 x 10-5 Torr was achieved. Targets were then heated to ~600 C in H₂ atmosphere at ~1 Torr pressure and allowed to cool. H₂ was released again by heating to 900 C. At least three cycles of H₂ absorption/desorption were given to create active sites for D₂ absorption.

After release of all H₂, the target was heated to 900 C in D₂ atmosphere at ~1 Torr pressure and allowed to cool by switching off the induction heater. D₂ gas was absorbed While cooling. At least three cycles of D₂ absorption/desorption were given similar to H₂ absorption/desorption. It was found that the absorption increased in each cycle and tended to saturate with 2nd or 3rd cycle.

Fall in pressure recorded by an oil manometer is a measure of the quantity of D_2 absorbed. It was noticed that each target could typically absorb ~1019 molecules of D_2 . Considering that mass of Ti is a few hundred milligrams, this corresponds to an overall D/Ti ratio of <10-3 only. However, since most of the absorption is presumably on the

surface, it is likely that the D_2 density is higher at the surface.

While preparing the targets, we found that their quality depends on various experimental factors. Some of them are listed here.

(i) Initial sandblasting of the targets for cleaning and roughening of the surface leads to better absorption of D.

(ii) Impurity content (such as O2, N2 etc) in D2 should be <0.1%.

(iii)Since the glass vacuum chamber is isolated from the pumping system during D₂ absorption, it is important that the vacuum chamber be leak tight. Small air leaks may contaminate the D₂.

Evidence for Production of Tritium via Cold Fusion in Deuterium Loaded Palladium

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After the first announcement reporting the observation of cold Fusion (1, 2) lot of evidence supporting the same have appeared in scientific literature and Press. Palladium an titanium loaded electrolytically (1,2) and titanium loaded directly with deuterium gas (3) have been reported to emit neutrons. However not many references have dealt with tritium measurement. Neither gas loading experiments involving Pd-D₂ nor tritium measurements in this have been reported. Experiments have been carried out in our group in the Pd-D system and tritium measurements in this system. The results obtained so far seen to strongly support the occurrence of Cold Fusion reaction.

EXPERIMENTAL

Preparation and pretreatment of Pd samples: For loading deuterium gas in Pd, two types of samples were used. One was Pd-Ag alloy supplied by M\s. Johnson-Mathey as directly used without further surface treatment. These were subjected to 600 c and vacuum of better than 10-5 mm or better for prolonged period, before use. The Pd black powder was prepared from PdCl₂.

Preparation of deuterium gas The D₂ gas used for gas loading as prepared from D₂O (supplied from production of HWP(B) and having a tritium activity of 0.075nci\ml) reducing with Na in a high vacuum system under stringent conditions. The gas produced gas is stored in a SS cylinder pressurising it with Activated charcoal liq N₂ cooling. The gas thus produced was not further analysed for tritium as it is expected to contain only

0.038nci\l activity.

Loading D₂ gas in metal phase: The schematic drawing of the experimental set up used for gas loading is given in Fig 1. It essentially consists of a vacuum system equipped with a rotary pump and an oil diffusion pump giving a vacuum of 10—6 torr. The reaction vessel C containing Pd sample is connected to the vacuum system through a buffer tank B. Deuterium cylinder D is connected to the vacuum system through needle valve V1. The entire system is of SS and tested for a vacuum of 10—6 torr and pressure of 100 Kg.cm—2. The system is also equipped with a pressure guage G and a manometer / pressure guage VG. A weighed amount Of Pd (black or metal) was taken in the vessel and heated to 600 C for 2 hrs. After cooling to room temperature, deuterium was filled at 1 atm. pressure and allowed to attain equilibrium.

Preparation of Samples for Tritium Analysis: The vessel containing Pd sample was transferred into a closed glass container in a dry enclosure, free of moisture and O₂ and equilibration as kept at few hours to over night. This was necessary, as D₂ absorbed Pd samples hen exposed to moisture or O, is known to catalyse the recombination of absorbed D₂ gas with O₂, with considerable increase in temp. The entire deuterium absorbed is found to be desorbed through this step. Tritium counting —Liquid samples containing tritium were analysed in a liquid scintillation Spectrometer, for varying time and applying different corrections, as per procedures given in a separate paper else where in this Report. Autoradiography of the deuterium loaded samples. The experimental procedure for this investigation is described elsewhere in this report. It essentially consists in keeping. The gas loaded sample on negative Xray film and developing them after predetermined time

Results and discussion

As expected, absorbing of deuterium in pd-black as very fast and it readily gave a

stoichiometry of pd D 0.6 (as calculated by reduction in pressure of deuterium). But in case of the alloy the absorption as rather slow and also the amount of deuterium absorbed

as much less than that corresponding to a bulk Pd D 0.6.u

As mentioned earlier we employed equilibration with distilled water for determination of tritium. This method was chosen for its simplicity and rapidity. Thy other possibility would had been to liberate the absorbed deuterium gas completely at 600° C and then either counting it directly in ionization chamber or oxidizing it to corresponding oxide and than analysing by liquid scintillation counting. But this becomes very impracticable because of the low amounts of gas absorbed (in the small amounts of metal samples taken) Taking into consideration the exchange reaction.

Tabs + H20 = Habs + HTO(1) and applying law of mass action one obtains following relation for low tritium concentration:-

Yi.na = nl[xe(1/k + 1) - xi]....(2)

where x & Y refer to tritium atom fraction in the absorbed and the liquid phase respectively and subscripts i and e refer to initial and equilibration conditions respectively. na & nl are the gm moles of gas absorbed in metal & gm moles of water taken for equilibration

K is the equilibrium constant for reaction (1) and was taken to be same has for exchange of tritium between hydrogen and water, as after equilibrium the system consists mainly of H both in the absorbed and the liquid phase. The value of K is 6.128 at 30°C [Ref.4].

Yi calculated from equation gives the No of tritium atoms produced for every deuterium atom absorbed in the metal. The tritium atom fraction x can be calculated from the tritium activity A applying equation 3.

1
$$x \circ =$$
 $d \times 3320$
where A is activity in ci/ml and d is density of water in gm/ml.

$$(d = 0.996542 \text{ at } 27 \text{ oC})$$

The results of two typical experiments conducted are given in Table (1)

The samples of deuterium loaded metal were also subjected to autoradiography [Ref.6] for direct evidence of tritium. In case of Pd-black powder no fruitful conclusion could be drawn as the x-ray film got spoiled because of the powder sticking to the film. But in case of Pd-Aq soils it gave unmistakable fogging of x-ray film once again indicating the emission of some radiation from the foils

Unlike the Ti-tritium system the emission of Pd-K α does not happen in the case of Pd-tritium system, as is known.

The Pd samples loaded with deuterium therefore did not show any thing (Ref.7). But one important observation made in this case where Pd foils ere loaded with deuterium after using the system for Titanium sponge. The titanium sponge, though had absorbed substantial amounts of deuterium (corresponding to Ti Du 1.7), had not shown any signature of tritium. But the Pd samples loaded subsequently in the same cell ere exhibiting $K\alpha$ of Ti. The surface of the Pd foils was then found to be contaminated with Ti. This was another indirect proof of tritium having been produced in the Pd-deuterium system.

There is a clear cut evidence for production of tritium in the Pd-D2 system and they

most plausible explanation appears to be fusion of two deuterons giving tritium.

The system did not require high pressure of D₂ gas also no special efforts ere made to creates no non-equilibrium conditions as suggested by De Ninnoetal (Ref). Although no comparison between the electrolytically loaded & gas-loaded Pd can be made as yet; electrolysis does not seen to be a necessary prerequisite for inducing cold fusion. However a comparison in terms of 'yield' of cold fusion and also the mechanism of cold fusion in the two system would be interesting. Unfortunately no so far neutron measurements have been carried out so far in the present work. A correlation between neutron production if any & tritium production would contributes significantly towards confirming the mechanism being cold fusion.

Acknowledgements: — Authors sincerely wish to express their gratefulness to Dr. P.K. Iyengar, Director, BARC for his keen interest and constant guidance in the present work. We are also thankful to Dr. M. Srinivasan for fruitful discussions. We also thank Dr. A Shyam and Mr. R.K. Raut for autoradiography and Dr. Madan Lal for the Si(Li) detector measurements.

Dr. T.S. Iyengar has carried out Tritium measurements described in this paper, which has been included elsewhere in this report. The authors would like to express their thanks to him.

Shri. V.H. Patil has been of great assistant in the experimental work. We thanking for same.

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TABLE 1
Summary of Results of Gas Loading Experiments in Palladium

Sl.	Sample	D/Pd Ratio	nl/na	Tritium activity of water used for equilibration, Xi	Tritium activity of water after equilibration	Yi T/D ratio in metal
1.	Pd-black	Ø.63	46.96	1 pci me-1	Ø.22 nci/ml	3.615x10-12
2.	Pd-Ag foils	Ø.46	213.33	-do-	Ø.16 nci/ml	12.63x1Ø-12 (1.263x1Ø-11)

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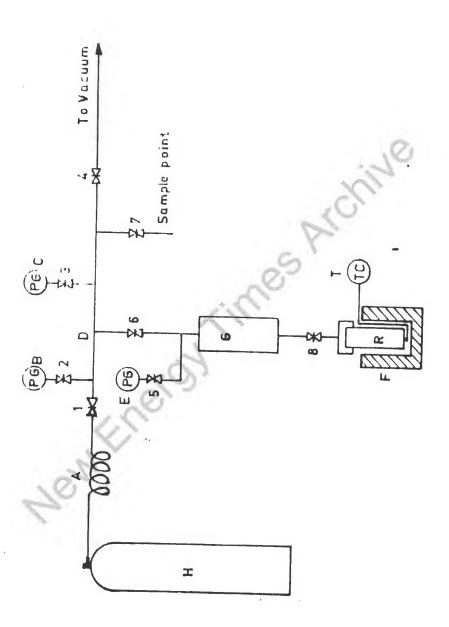


Fig.1. Schematic diagram of high pressure equilibration system

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MATERIALS ISSUES IN THE SO CALLED "COLD FUSION" EXPERIMENTS

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In a recent electrochemical experiment with palladium cathode and platinum anode, immersed in 99.5% D₂O + 0.5% H₂O plus 0.1 M LiOD, M. Fleischmann and S. Pons¹ claimed to have seen an "excess heat" which they ascribed to "cold fusion" of deuterium nuclei electrochemically infused into the palladium lattice. These authors and Jones et al² have attempted detection of neutrons/H³ as signatures of fusion based on the well known reactions:

 $D + D = He^3 + (0.82 \text{ MeV}) + n (2.45 \text{ MeV}),$ $D + D = H^3 + (1.01 \text{ MeV}) + p (3.02 \text{ MeV}),$

and have found some favourable evidence. So far efforts to confirm their findings made in various laboratories, including BARC, have been somewhat inconclusive, although some neutrons appear to have been seen occasionally. The reported neutron production is about six to nine orders of magnitude less than what the "excess heat" would imply. These observations have generated a great amount of debate concerning the nuclear physics of D-D reaction; novel ideas have been proposed including the reaction $D + D => He^4$, with the added requirement that the energy (23.8 MeV) be delivered directly to the lattice. Even when they have been observed, often it has been claimed that neutrons appeared only intermittently. In view of these features we deem it prudent to draw attention to some of the relevant solid state aspects of Pd-D system which might be involved in some way.

Absorption of hydrogen/deuterium by Pd has been studied for a long time and is known⁴ to be strongly exothermic. Neutron diffraction⁵ experiment shows that H/D goes into the octahedral sites in Pd lattice. Accompanying this uptake is a structural change with the solid changing from the α phase at low H/D concentrations to $\alpha + \beta$ phase at higher values to an eventual β phase. Both the phases are f.c.c. with cell constants 3.89 A and 4.03 A respectively. Calorimetric studies⁴, using activated Pd and molecular D[-]2[-] gas, show that at 30° C, the heat released during the formation of PdD_x, rises from ≈ 7.50 K Cals per mole of D₂ for $x \approx 0.023$ to ≈ 8.43 K Cals per mole of D₂ for $x \approx 0.422$, with the authors asserting that "these heats show a definite increase for each increment of gas added throughout the mixed phase region". Thus as more D atoms are loaded into Pd, one may expect that the β phase regions (with a higher lattice constant and presumably better cohesion) would grow in the matrix of α phase, leading to regions of localized strains. We may conjecture that eventually abrupt atomic readjustments may occur giving rise to conditions—such as local heating and energetic deuterium motions—that are relatively more favourable for some of the D-D fusion mechanisms proposed in the literature. This description also suggests that the results of the electrochemical experiments could be very much sample dependent.

Let us next turn to the enthalpy release. Actually there are several adsorption, absorption and desorption processes involved in the experiment using an electrochemical cell. But here we will concentrate only on the heat of formation of PdD_x , which has not received enough attention. We first note that in electrolysis, using Pd as a cathode because of its special ability to dissolve H/D, one generally does not observe any evolution of H_2/D_2 at the beginning, as these are absorbed by Pd. Also we may bear in mind that the values of the enthalpy release in the formation of PdD_x quoted above from ref. 4 relate to the situation with molecular D_2 . If we were to measure these using nascent (i.e. atomic form) deuterium, then the enthalpy released would be larger by the dissociation energy⁶ of D_2 , viz 106 K Cals per mole of D_2 . Assuming that the (cathodic) current in the Fleischmann — Pons experiment is only due to flow of D^+ , we can then infer

BARC Studies in Cold Fusion

the amount of deuterium impinging on the Pd cathode and then roughly estimate the rate of enthalpy release due to deuteride formation. We estimate that it is of similar magnitude as the claimed "excess heat" by Fleischmann and Pons and emphasize the need to include it in the total energy balance calculations.

To sum up, we feel that the neutronic signals reported to have been seen in some of the recent electrochemical experiments deserve to be viewed in the light of the materials science of palladium deuteride. Although if it is finally confirmed this so called "cold fusion" would be physically very interesting, the possibility that it will lead to a significant new energy source appears doubtful at present.

We acknowledge helpful discussions with a large number of colleagues especially Dr. P. K. Iyengar, Dr. S. Gangadharan, Dr.T. P. Radhakrishnan and Dr. S. K. Sikka.

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MATERIALS CHARACTERIZATION SECTION



Relatively little emphasis has been placed on the role of materials in "cold fusion" experiments and generally characterization of the anode and cathode materials has not played a significant role in the discussions. Most experiments have utilized Pd cathodes for the electrolytic charging and Ti (or Ti alloys) for gas charging experiments following the examples of the initial experiments by Pons, Fleishmann, and Jones. In addition, most electrolysis experiments have utilized Pt anodes, with the important exception of those at Texas A&M which are reporting ³H generation and which utilize Ni anodes.

It has become increasingly common for the D/Pd ratio attained to be reported in the electrolysis experiments with the measurements generally being based on the weight gain of the cathode. Values of D/Pd in the range 0.7 to 1.1 have been commonly reported for experiments with both positive and negative "cold fusion" results. The gravimetric method of measurement is generally relatively reliable for D/Pd values in the range of 0.6 to 1 as long as the loss of D is minimized between the period of charging and the weighing. Most investigators do not report the details of their procedures, such as the storage of specimens at low temperature to avoid this problem. An additional difficulty in these experiments is that many investigations report deposits on the cathode or diffusion of Li into the cathode and these must be removed before weighing to determine the D/Pd values. It does not appear that this has been done.

In a number of cases the cathodes have been analyzed for ³H, ³He, and ⁴He with negative results. Reports of diffusion of Li into the cathodes have been made but the bases for these reports have generally not been specified. In one case the presence of Li up to several micrometers into the Pd cathode was detected by SIMS measurements but the authors suggest that this was the result of surface cracking and contamination of the crack surfaces by the electrolyte. They also detected other components of the electrolyte in the cathode by the same technique.

Even less characterization of the materials has been carried out for the gas charging experiments. Often mixtures of metals have been used and they have been characterized as "turnings", "sintered powder", "mossy solids" etc. none of which have any meaning with respect to the materials character. The D/Pd ratios have not been determined nor has sufficient information been given to even allow an estimate to be made. This is particularly true as the absorption of D in Ti and its alloys is a very surface sensitive process.

As discussions of the success or failure of "cold fusion" experiments have often been ascribed to properties of the cathode materials it is useful consider the various parameters suggested to be important in light of what is known of the behavior of D(H) in Pd and Ti.

a. Success of the electrolytic experiments has been ascribed to the very high "confinement pressures" achieved at the overpotentials applied. As discussed in Appendix -- the overpotential results in a very high fugacity of D at the Pd surface but this corresponds to a much lower pressure due to the non-ideality of D2 gas and to the loss of D2 by gas formation at the surface. The compositions attained by cathodic charging correspond to a very moderate pressure of 15 kbars. - 15 and - 15 and - 220,000

prospere inh.

b. The suggestion has been made that as a result of the high fugacity, confinement of D in Pd results in a very small D-D distance. As discussed in Appendix -- the smallest distances between interstitial sites occupied by D in Pd is 0.151nm which is large compared the D-D distance in the D₂ molecule. Significant dual occupancy of interstitial sites by D is not in accord with the linearity of the lattice expansion measured by x-rays or with theoretical treatments of the Pd-D system; smulting show-let dual crapping will that yiel any smulting show-let dual crapping will that yiel any smulting show-let dual crapping will that yiel any smulting show-let dual crapping will the sound to the same state of th layor than that sa

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c. The suggestion has been made that successful experiments require very long charging times. As discussed in Appendix -- the diffusivity of D in Pd is sufficiently large at 300 K so that equilibrium can be attained in a 1 cm thick sheet of Pd in times of the order of hours.

- d. A suggestion that a particular form of the cathode material, e.g., cast material, must be used to minimize the dislocation concentration is not in accord with known behavior of Pd as discussed in Appendix --. The very large inhomogeneous deformations which accompany cathodic charging of Pd under the conditions used result in high dislocation densities even in a well annealed material. Thus regardless of the initial starting point, the charging conditions result in severe deformation of the cathode.
- e. Deposition of various materials from the anode and from the electrolyte (leached from the container) on to the cathode has clearly occurred in many of the experiments. Analysis has revealed Pt (probably from the anode), Si (probably leached from the glass container), Cu, Zn, Fe, Pb, and other trace elements. The surface contamination may affect the rates of reactions at the cathode surface and therefore the effective fugacity. In general surface poisons increase the fugacity by decreasing the rate of D₂ formation. This is indicated by the high D/Pd values attained and by the observation, reported in several experiments, that the loss of D from the cathode after electrolysis was slow. Thus surface contamination probably had the effect of increasing the D/Pd values and the fugacities attained. "Dendritic" growths were reported on the surfaces of the cathodes in the Texas A&M experiments which utilized Ni anodes. This has lead to the suggestion that these dendrites are of significance, possibly due to increased electric fields at the "dendrite" tips, in the formation of ³H in the electrolyte. Other experiments which reported these "dendrites" have not reported generation of ³H. It is difficult to understand the importance of these "dendrites" particularly as these growths are not uncommon in plating experiments. The suggestion that cans of helpost every con he crestile as a result of the ortin of the destrite is wirest.

APPENDIX --: MATERIALS FOR COLD FUSION

A. The Palladium - Hydrogen (Deuterium) System

"Cold fusion" experiments have utilized two types of cathode materials; palladium and titanium (including several titanium alloys). These two metals have significantly different behaviors in the presence of hydrogen and its isotopes. Their response to exposure to hydrogen isotopes has been extensively studied and well documented^{1,2}; particularly in the case of palladium. In the following, we shall highlight some of the behavior of the Pd-H and the Ti-H systems selecting those aspects which are most pertinent to the issues raised by the suggested "cold fusion" experiments. Much of what is known derives from experiments using the mass one isotope, ¹H (also denoted by H), but studies have sufficiently established the isotopic dependence to allow the behavior of deuterium, ²H (also denoted by D), to be deduced where measurements on D are not available. Many aspects of the behavior of hydrogen isotopes in bcc and fcc (Pd) metals have been clearly shown¹ to be dominated by their behavior as quantized particles; other properties can be understood using classical concepts. Behavior as quantized particles has not been established for hydrogen isotopes in the hcp crystal structure (titanium) but such behavior may be expected.

Palladium has a face centered cubic (fcc) close packed crystal structure and as hydrogen is added this structure is maintained across the phase diagram³. It can absorb large quantities of H(D) with concentrations as high as H/Pd=1.1 being attainable. The H(D) occupies predominantly octahedral interstitial sites which if they were all filled would result in a H(D)/Pd ratio of 1. Hydrogen can also be accommodated in tetrahedral interstitial sites at a somewhat higher energy and it is believed that these sites may be partially occupied at the higher H(D)/Pd ratios. The spacing of H(D) occupying these sites is not particularly small as shown by the table below. Both the octahedral and tetrahedral sites have cubic site symmetry and since H(D) appears to have random occupancy in the occupied sites the

DEUTERIUM - DEUTERIUM DISTANCES

SITES OCCUPIED	DISTANCE IN NANOMETERS
nearest neighbor octahedral sites	0.151
nearest neighbor tetrahedral sites	0.275
nearest neighbor octahedral - tetrahedral sites	0.168
vacancy sites	0.185
molecular deuterium gas	0.074
liquid deuterium	0.27 (jut olso 0, 074)
water	0.151

maintenance of cubic crystal structure is consistent with the high H(D)/Pd values attained. Further evidence for the random occupancy of sites and the cubic distortion of the lattice is provided by x-ray studies of the Pd-H(D) system⁴ which also indicate that the behavior of D is closely similar to that of H. Recent theoretical treatments^{5,6} of the behavior of H(D) in

3

Pd are consistent with the conclusion that H(D) does not attain exceptionally close nearest neighbor distances even under dynamic conditions. Molecular dynamic simulations⁵ at concentrations up to D/Pd = 1.1 found no D-D distances shorter than 0.07 nm compared to the molecular D-D distance of 0.074 nm. Interactions of D with lattice defects have been studied both experimentally and theoretically. Significant attractive interaction energies are found⁷ with those defects which have a decreased electron density such as vacancies. Palladium has such an attractive interaction but it is particularly weak. Each vacancy can accommodate up to 6 D interstitials with the D solutes occupying sites displaced towards the octahedral interstitial sites adjacent to the vacancy. The D-D distance is 0.185 nm.

The phase diagram of the Pd-H(D) system^{2,3} is typical of that of many of the bcc and fcc hydride forming systems. The dominant feature is a miscibility gap with a critical temperature of about 549 K and a critical composition of H(D)/Pd \approx 0.27. At about 300 K the initial α phase has a solubility of about H/Pd \approx 0.03 and the two phase region extends to about H/Pd \approx 0.65 at which point the β is formed. Both the α and β phases have fcc structures. Phase relations in the Pd - H system depend on the isotope of hydrogen used but the differences are not very large. Important differences exist in the P-C-T data^{2,3} which characterize the equilibrium of gaseous hydrogen isotopes with Pd but these differences are consistent with the differences in the isotopic masses. Many of the thermodynamic properties of the Pd - H(D) system are consistent with theoretical calculations based on mean field theory⁸ which is based on a repulsive nearest neighbor interaction between H solutes. This repulsive interaction has been measured using a variety of methods^{7,9,10,11}

While the α and β phases are solid solutions of H(D) in the Pd interstitial sites, formation of long range ordered structures does occur at high concentrations and low temperatures¹². These structures form by the ordering of the H(D) interstitials on subsets of the interstitial sites and are consistent with the nearest neighbor repulsive interactions between the H(D) interstitials. At H(D)/Pd = 1 the structure is of course ordered if the H(D) occupies octahedral interstitial sites. No other hydrides are known to form at temperatures of the order of 300 K in the high concentration region of the phase diagram although few careful investigations have been carried out in this region. The lattice parameter measurements⁴ which extend into this composition region indicate a quite linear lattice expansion up to H/Pd = 1.0.

Hydrogen and its isotopes diffuse very rapidly in the Pd lattice⁸; at 300 K the diffusivity of H in the α phase is about $2x10^{-9}$ m²s⁻¹. The diffusivity in the β phase is somewhat slower but is still very high. The effect of isotopic mass on diffusivity is very non-classical in Pd with the D_D > D_H > D_T at 300 K; a clear indication of the quantum mechanical tunneling process which dominates the diffusion of H(D) in the Pd lattice. With respect to the "cold fusion" experiments an important point is that with the known diffusivities of D in Pd, a time of the order of 3.5 hrs. is required to achieve equilibrium during the charging of a 1 cm thick sheet specimen.

Hydrogen charging of Pd can be carried out in several ways. Electrolytic charging with the Pd as a cathode has been carried out in a number of different electrolytes. In general, the Pd surface is highly active towards the dissociation of the H_2 (D_2) molecule and hence little H_2 (D_2) gas is evolved until relatively H(D)/Pd ratios are obtained at which point the back diffusion of H(D) to the surface becomes significant. Additions of "poisons" such as As

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ions to the electrolyte increase the fugacity of the H(D) at the surface by decreasing the formation rate of H_2 (D_2) and hence allow a higher H(D)/Pd value to be obtained with the same overpotential. Since the fugacity of hydrogen depends on the overvoltage as well as on the surface conditions it is difficult to obtain a direct relation between the cathodic charging conditions and the H(D)/Pd values obtained. Since H_2 (D_2) is a highly non-ideal gas, at high fugacities the equivalent pressures are many orders of magnitude less than the fugacities.

Gaseous charging of hydrogen is also commonly carried out¹³ and high H(D)/Pd ratios can be obtained for relatively moderate pressures. Solution of H(D) in Pd follows Sieverts law at low pressures and then shows significant deviations from ideal solution behavior, probably as a result of H(D) - H(D) interactions in the Pd. In the region of Sieverts Law behavior the proportionality of the H/Pd to the $P_{H2}^{1/2}$ is further evidence for solution of H(D) in Pd in the atomic form rather than as a molecule.

Concentrations of D/Pd \approx 1 are often attained in "cold fusion" experiments and are often quoted as a necessary condition. It has also been suggested that the "very high confinement pressures produced by electrolytic charging" are necessary for "cold fusion". Comparison of the results of gaseous charging with electrolytic charging allows an estimate of these "confinement pressures" to be made. A concentration of about D/Pd \approx 1 requires a gas pressure of about 15 k bars at 300 K¹². Thus the effective pressure corresponding to the high fugacities calculated from the overvoltages during cathodic charging are equivalent to a very moderate pressure indeed.

Palladium is an "exothermic occluder" of H(D), i.e. the heat of solution relative to the gas phase as a standard state is negative. The heat of solution is relatively small having a value of -19 kJ/mole H_2 in the α phase^{2,3} a value of about -46 kJ/mole H_2 in the β phase¹¹. Values for the heats of formation of the hydrides in the Pd - H(D) system have not been measured but most hydrides have heats of formation in the range -58 to - 209 kJ/mole H_2^2 .

Formation of the β phase from the α phase during charging causes a large amount of lattice strain and deformation as the phase change is accompanied by a large increase in volume. Since the molal volume of the β phase is larger than that of the α , the β phase is in compression during charging. Correspondingly, the plastic deformation of the α phase during charging coupled with the decrease of volume during loss of hydrogen during the β to α phase change causes very high tensile stresses at the surface during the loss of hydrogen. The net result is significant plastic deformation during charging and deformation and fracture during loss of H(D).

B. The Titanium - Hydrogen (Deuterium) System

While considerably less is known about the Ti - H(D) system than about the Pd - H(D), our knowledge is sufficient to answer many of the questions of interest for the "cold fusion" experiments. At about 300 K hcp titanium absorbs H(D) in the α solid solution up to concentrations of about H(D)/Ti \approx 0.05 although low temperature measurements are not very accurate due to the difficulty in absorbing H(D) from the gas phase at low temperatures. Above this concentration a two phase equilibrium exists between the hcp α solid solution and the γ hydride. This hydride exists over the composition range TiH(D)_{1.9}

to TiH(D)₂ having a flourite fcc structure at the lower compositions and a face centered tetragonal structure as the H(D) concentration is increased. At temperatures above about 570 K H(D) solubility is about H(D)/Ti \approx 0.1 and the solid solution is in equilibrium with the bcc β phase of Ti which is stabilized by H(D). The phase has a very high solubility for H(D) of the order of H(D)/Ti \approx 1. Above this composition a two phase region exists where the β phase is in equilibrium with the γ dihydride, TiH(D)2. Since the β phase of Ti can be stabilized by many other solute additions, it is possible to absorb large quantities of H(D) into the β stabilized alloys at 300 K without forming hydrides. The extent of this low temperature solubility in the β stabilized alloys has not been established.

Site location studies have not been carried out for the Ti - H(D) alloys. Random occupancy of tetrahedral sites in the non-stochiometric γ hydrides was deduced from NMR measurements¹⁴. No information is available for site occupancy in the α and the β phases. If we assume tetrahedral site occupancy in the hcp α phase and both octahedral and tetrahedral site occupancy in the β phase the nearest neighbor distances given in the table below can be calculated.

DEUTERIUM - DEUTERIUM DISTANC	CES IN TITANIUM
α PHASE (HCP)	D - D Distance in nm
nearest neighbor tetrahedral sites	0.23
γPHASE (FCC)	
nearest neighbor octahedral sites	0.22
β PHASE (BCC)	
nearest neighbor octahedral sites	0.17
nearest neighbor octahedral-tetrahedral sites	0.083

The thermodynamics of the Ti - H(D) system has been extensively studied. Formation of solid solutions of H(D) in the α and the β phases are exothermic as is the formation of the γ hydride. Differences of these values between H and D are very small. These enthalpies are given in the table below^{2,15,16,17,18,19}.

Measurements of diffusivity of H(D) in Ti have not been carried out but the diffusivity at 300 K can be deduced from permeation experiments to be about $1x10^{-10}$ m²s⁻¹. This diffusivity would allow equilibration of a 1 mm thick sheet with a H₂(D₂) atmosphere in

ENTHALPIES OF SOLUTION AND FORMATION (kJ / mole H2)

	HYDROGEN	DEUTERIUM
solution in α phase	-90.4	-94.6
solution in β phase	-116.4	
formation of γ phase	-138	-121

0.7 hrs. Diffusion or permeation experiments are difficult to perform at low temperatures due to the presence of surface oxides on the titanium which inhibits the entrance of H(D) from the gas phase. Equilibration of Ti with gaseous H₂ is slow due to surface oxide. Even under cathodic charging conditions entry of H(D) is inhibited unless the surface oxide is minimized and "electrolytic" poisons used. As a consequence equilibration in a gaseous atmosphere or under cathodic charging is dominated by surface reactions.

Titanium hydrides are brittle and undergo cleavage when stressed. They serve to embrittle Ti and its alloys, causing materials which are only partially hydrided to powder. The introduction of H(D) causes a large volume increase as a result of the molal volume of solution (about 0.28 of the Ti atomic volume). In addition, formation of the hydride causes a volume increase of about 23% as a consequence of the difference in H(D)/Ti values for the α and γ phases and the volume increase accompanying the crystal structure change. It is not surprising that the gas charging "cold fusion" experiments report fracturing of the Ti specimens used.

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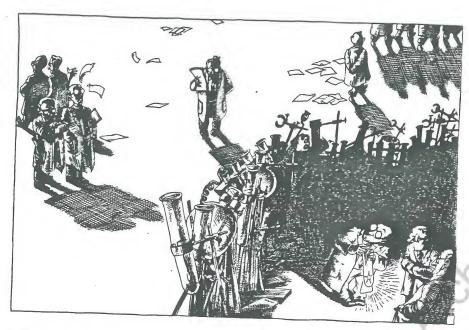
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SCIENCE AND TECHNOLOGY



The secret life of cold fusion

PALO ALTO, CALIFORNIA, AND CAMBRIDGE, MASSACHUSETTS

Cold fusion disappeared from the public eye with striking speed, but many scientists are still working on it. The field frustrates and demoralises those who want easy certainties. At the same time it provides a fascinating example of the sort of chaos from which scientific certainties are born

SIX months ago cold fusion burst upon the world. Three months ago it had vanished from sight. The popular wisdom is that nothing was demonstrated except credulousness and sloppy experimental technique. However, a coterie of scientists, respected experimenters among them, stubbornly insist that cold fusion happens, and that it generates energy—albeit sporadically and in unpredictable amounts. Be it gullibility on a grand scale, a violation of the laws of nuclear physics, or deviant chemistry, the "cold fusion" phenomenon still needs explaining.

The number of laboratories involved has shrunk since the heady days of April. Many teams ran out of patience and money, yet believers and some sceptics struggle on. A dozen or so groups say that their apparatus produces energy. Some have found tritium, a heavy type of hydrogen that is produced by nuclear reactions, and some have spotted radiation. Others have come up with lots of reasons to doubt whether anything is going on at all.

Whatever the outcome—and it may not be known for years—the dispute has become a classic scientific controversy. Theories, reputations and money are at stake. Scientific fact has become elusive; for fusioneers, faith is essential. Every claim they make can be questioned, yet every doubt cast on them can, it seems, be shrugged off. Everything is still to play for.

The story so far

On March 23rd Dr Stanley Pons, of the University of Utah, and Dr Martin Fleischmann, of Southampton University, held a press conference to unveil a new source of energy. They were both respected practitioners of electrochemistry, the study of chemical reactions driven by electric currents. Their apparatus seemed childishly simple; a Utah fusion cell is little more than a test tube fitted with two electrodes, one of them made from the rare metal palladium. The test tube contains heavy water. Where normal water has atoms of hydrogen, heavy water has deuterium, a heavier form of the

element.

The deuterium is the crux of the matter. Dr Pons and Dr Fleischmann claimed that pairs of deuterium nuclei inside or on the surface of the palladium were joining together to make heavier nuclei. This fusion process is well known to give off energy—it drives stars and hydrogen bombs. But it normally requires the nuclei, which repel each other, to be brought together with great force. The Utah claim caused uproar because Dr Pons and Dr Fleischmann claimed to be seeing fusion with un-energetic, roomtemperature nuclei—hence "cold fusion".

It was clear that Utahn cold fusion broke the laws of physics. Breaking laws is the way to find better ones; it's fun to boot. Within days, the best physics laboratories in the world—as well as the second-best, and quite probably some that are no good at all—were trying their own experiments. So were the world's electrochemists. Early experimental confirmations amounted to little, especially since some were quickly followed by retractions, which of course fuelled scepticism. Dr Pons and Dr Fleischmann grew reticent, and unhelpful to sceptics.

Much of the early research was freelance, with money and equipment siphoned off from other projects. After a few months, many part-time fusioneers had to get back to the jobs they are paid for. Some new sources of money have since opened up. In America. the Electrical Power Research Institute, which raises money from the energy industry, has allocated \$800,000 to research this year. According to Mr Bindi Chexal, a programme manager who oversees the institute's fusion budget, \$2m will be available next year. The state of Utah, flushed with local pride, has put about \$5m aside for fusion research. Japan has set up an 80-strong fusion research institute. India is devoting the resources of two of its national laboratories to fusion research. There are similar programmes in other third-world countries. Meanwhile corporations have been keeping a close eye on what is going on. Scientists from America's General Electric have been frequently spotted around the University of Utah. Some are visiting Dr Pons this week.

Alarums and excursions

The people still working on the problem—the hard-core fusioneers—have reached a degree of consensus. They believe that fusion cells can behave in more than one way. They can give off tiny amounts of tritium and a few neutrons, but no heat; they can give out steady heat for a few days; or they can give off intense bursts of heat.

The first sort of behaviour, products-

without-energy, was identified by Dr Stephen Jones of Brigham Young University, also in Utah. Similar effects have been seen in numerous laboratories, including Los Alamos National Laboratory in New Mexico. No explanation for them is universally accepted, but nor is anyone unduly worried by them. If this was the only sort of cold fusion on offer it would be a minor scientific curiosity.

It is the phenomena that involve energy that arouse passions. In some laboratories, cells give off energy (in the form of heat) 10-50% faster than energy is put into them (in the form of electric current). Laboratories at Texas A&M University, Stanford in California, Utah, Florida, Minnesota, Tennessee, in Italy and in India have claimed this sort of result, to cite the most prominent.

The third type of fusion is the "heat excursion". After weeks at a steady level, the cell's temperature suddenly climbs to hitherto unapproached heights, stays there for hours or maybe a day, and then returns to normal. This sort of day-trip fusion has been seen mostly by the two independent groups in Utah. Heat excursions have an energy output up to ten times the energy input. They stop as suddenly as they start. Nobody knows why.

As well as heat, there is tritium, another type of heavy hydrogen. Two groups at Texas A&M University, one led by Dr John Bockris and the other by Dr Kevin Wolf, have detected tritium in fusion cells. With tritium and heat, one can construct an argument. If heat is being produced, it must come from somewhere. The same is true of tritium, which is radioactive and shortlived, and thus rare. A deuterium fusion reaction can produce heat and tritium, so it offers a simple explanation of the facts. Unfortunately it does not explain all the facts, it contradicts some other facts, and to accept it you must junk large amounts of atomic

Test-tube fusion

Palladium

physics that have been gospel for 50 years.

It may be easier to get rid of the facts than try and make sense of them. There is little doubt that people are measuring tritium; but it is possible that there is tritium in the palladium before the experiment starts. Dr Wolf is now doing

The deuterium goes towards the palladium electode

The electric current solits he

experiments that carefully compare the tritium in the electrodes before and after.

In the case of the heat there are some who do not believe it exists at all. The trouble lies in calorimetry, the measurement of heat. Everyone used to think calorimetry was straightforward. Now that so much rests on it, scientists have rediscovered its awkwardnesses. Dr Nathan Lewis of the California Institute of Technology has looked at some of them. The most fundamental is the calibration of the equipment. Since the amount of liquid can change as some bubbles away, the relationship between heat and temperature can change. The ramifications of this simple point make heat output hard to measure. The fusioneers' tendency to leave their cells open to the air makes matters still worse. According to Dr Lewis, no one who uses sealed cells has reported seeing heat.

Dr Walter Myerhof, also of Stanford, has tried to work out error margins for fusion-cell calorimetry. He concluded that it is reliable only to within 15-20% of the value of a given reading; enough to account for the constant low-level heat output measured by many laboratories. What then about the large energy output during heat excursions? Dr Lewis points out that such short-lived phenomena could be due to fluctuations in the chemical conditions of the experiment. The fusioneers admit that large quantities of heat sometimes get produced by such things, but claim to distinguish between these and "genuine" excursions.

Dr Lewis insists that no groups have properly shown a net output of heat over the entire length of an experiment. Remember, the cells have usually been absorbing electrical energy for long periods before giving out a burst of heat. They may be hoarding the energy away piecemeal, and then letting it all out at once. The technical term for a device that stores and releases electro-

chemical energy is not "fusion cell". It is "battery"

In response, fusioneers claim safety in numbers. They

electrode argue that, though errors can creep into any set of measurements, the chances are remote that a dozen different laboratories, using various types of calorimeter, should all be mak-(+) either one tritium nucleus If two deuterium nuclei undergo fusion, they \oplus may produce r one heliumthree nucleus

ing the same mistakes all the time. They also point out that heat does not seem to be generated when deuterium is substituted by ordinary hydrogen. Everyone agrees that fusion is inconceivable without deuterium.

One thing that would help convince outsiders would be a fusion cell that performed on demand. At present, the cells are capricious; their unpredictability fuels doubts and prolongs investigations. One of the problems the fusioneers have had is that their experiments last far longer than the media's attention span.

According to Dr Savarian Guruswamy, who works with Dr Milton Wadsworth's team in Utah, a typical cell will see a heat excursion once or twice every couple of months. In one six-week experiment, six cells produced heat once and one of them twice. Dr Bockris's group at Texas A&M saw low-level heat in four out of eight cells over three months. All 12 cells alongside produced tritium. Dr Glen Schoessow at the University of Florida claims to have seen low-level fusion in one cell for 32 consecutive days. It was still giving out heat as this page went to press.

The trouble with tritium

The debate over facts takes place on the fusioneers' home ground; they control the laboratories. It may end with the gradual admission of systematic errors, or with the development of cells that can be made to start and stop working on command. Even then they will need some theoretical framework if they are to convince outsiders. That could mean forcing electrochemists to make room for new chemistry. But if they are to prove nuclear fusion is going on, it will mean fighting on the critics' home ground: theoretical physics.

Physicists find cold fusion doubly impossible; impossible because of what does happen, and impossible because of what does not happen. When two deuterium nuclei fuse in the sun, they produce either a tritium nucleus and a proton, or a rare type of helium and a neutron (see diagram). Theory predicts that both helium and tritium should be produced in roughly equal amounts. Thus, besides tritium, researchers should be able to detect light helium, neutrons, and x-rays given off by fast protons.

They do not.

In one particularly damning experiment Dr Michael Solomon, a physicist at Utah, set up detectors in Dr Pons's laboratory. They ran continuously for six weeks, with the exception of a 24-hour hiatus when the university was struck by lightning. At the end of the six weeks, Dr Solomon had seen nothing, though the cells had been producing a little heat. Dr Pons told him that there had in fact been one heat excursion. It had taken place the day the detectors had been out of commission. Scientists who know Dr Pons and Dr Solomon say that relations be-

To Vo Cust Swed Fax: -Volve matic series □The tween the two have soured.

There is a little hope for fusioneers. The belief that tritium and helium should be produced in equal amounts stems from experience with nuclei free to collide at random. If the reaction takes place inside the palladium, as most fusioneers believe, then one set of products might possibly be favoured over another because the atoms in solid palladium are aligned in a particular way. Research by Dr Wolf and Dr P.K. Iyengar of the Bhabha Institute in Bombay suggests that this might be so. Independently, both groups measured that a fusion cell produces about ten million times as much tritium as helium. Their close agreement is far from conclusive. It is intriguing.

Dr Wolf calculates that if the Utahn cells were producing tritium at the same rate as his Texan cells, then the energy given off would account for the low-level heat seen by Dr Pons. Unfortunately the levels of tritium measured by Dr Pons are thousands of times lower than those measured by Dr Wolf, and by the team led by his colleague Dr Bockris. To make a dent in the physicists' disbelief, they need to find similar tritium levels in cells producing similar amounts of heat. They also need to demonstrate the relation between heat production, tritium and helium production, and radiation—the sort of correlation that Dr Solomon could not find in Dr Pons's laboratory.

There is a further theoretical problem with tritium. Theory predicts that the tritium created in deuterium fusion comes away from the reaction with a great deal of the energy that has been released. In fact, it has so much energy that it should react with any other deuterium nuclei it comes across. This second reaction would, in turn, produce its own characteristic products. None has been seen. Dr Wolf has calculated that if such a secondary reaction is to be avoided, then the tritium must be dumping the energy as soon as it gets it—but how and where, no one has the least idea.

Clashing by night

Suppose the fusioneers are wrong, and the heat comes not from nuclear fusion, but from a chemical reaction. How does that explanation stand up? None too well.

The obvious chemical reaction to invoke is one between oxygen and deuterium. The electric current in the cell tears heavywater molecules into their constituent deuterium and oxygen atoms, which takes energy. If those atoms then recombine to make water, they will release energy. So the heat might be explained by deuterium and oxygen recombining.

However, fusion cells can produce a lot of heat. After one of Dr Pons' heat excursions, his rough calculations—too rough to be accepted by his critics as reliable—showed that the cell had produced 8m joules of energy, enough to light one bar of an elec-

tric fire for a little under two hours. That much energy might have been produced if all the water molecules broken apart had recombined. But an electrochemist would expect no more than 3-5% of the separated oxygen and deuterium to recombine: not nearly enough. Dr Pons concluded that there must have been a net production of heat from the experiment—either due to a nuclear process, or some chemical reaction other than recombination. It would help his point if his calculations were more thorough.

Just as nuclear reactions leave tell-tale products, so do chemical ones, in the form of new molecules. Yet there have been no chemical changes measured in the palladium electrodes or the heavy water. In one experiment, Dr Bockris's group passed a small electrical current through the palladium to see if its resistance changed when



A simple experiment

energy was produced. A chemical change should cause a change in the resistance. None was found. Others have examined their electrodes under electron microscopes to see whether the fine details of their structure have changed, and found nothing.

There is no intrinsic reason to prefer unknown chemistry to unknown physics as an explanation, and neither is satisfactory. Therein lies a good reason to reject the heat measurements: it is more sensible to reject sporadic and dubious readings than to reject well-established theories. Were it not for the presence of tritium the fusioneers would probably have given up.

Many laboratories have done so already, unable to find a thing. But absence of evidence is not evidence of absence, and negative results are as open to doubt as any others. Take Britain's Harwell Laboratory. Little work on tritium could be done there because the source of the heavy water was a

nuclear reactor, which means it was inevitably contaminated by tritium. Some people at Harwell have told the fusioneers that they did see some heat and some neutrons but that the readings were low and unreliable. To those who desire certainty, that is not enough. Yet those working on new phenomena find it notoriously difficult to produce statistically significant work. And without statistical significance, it is hard to get novelty into scientific journals.

One team claims to have the results the world is waiting for. Dr Glen Schoessow and Dr John Wethington, two elderly scientists doing their research in the University of Florida's nuclear engineering department, say they can switch cold fusion on and off at will and control the amount of heat produced. Furthermore Dr Schoessow says that he has detected tritium, neutrons, and gamma radiation. He has not been able to match the rate of production of fusion byproducts to the rate of heat generation.

The two scientists and their patent lawyers are playing things close to their chests. Their laboratory is barred to prying eyes; they give few details about their work. Some who have seen the experiment are impressed. They report the intriguing detail that Dr Schoessow uses button-shaped electrodes unlike those of any other fusioneer. But a fusion group downstairs in the same building has disowned the two scientists.

Is nothing sacred?

When it was unleashed on the world cold fusion was described as simple. It depended on tried and tested techniques. Measuring neutrons and radiation, while not easy, is bread and butter to nuclear physicists. Calorimetry and electrochemistry were old tame disciplines. Yet even the most standard scientific procedure is open to objection when a new phenomenon is at play. All experimenters have their own style, their own ways of doing things. When there is no controversy such differences matter not a bit. Controversy makes everything open to question.

The niceties of measurement, normally taken as read by all concerned, become vital. They are scrutinised, and scrutiny brings to light complexities normally taken for granted, which in abnormal circumstances become matters of pressing concern. Only someone with hard-won experience can defend all the details of his calorimetry, or his electrochemistry, or his nuclear physics. Noone can defend all three, since there has never before been reason to master all three. So there is indefinite room for special pleading in the gaps between the disciplines. Only time can bring a unified expertise, and the hindsight to explain the erroneous results with which the field is doubtless littered. What, if anything, will survive that scrutiny must remain to be seen.

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-R.L. GARRIN-

September 29, 1989

TO: Members of the Cold Fusion Panel

NOW ELLON

Enclosed for your information is an article from the New York Times, a Special Call for Recent New Papers on Cold Fusion, a letter from Bockris to Bigeleisen and his letter to Kevin Wolf, an article entitled "Brookhaven Chemists Induce Fusion by Putting the Squeeze on -- With Clusters", and a Memo from Bigeleisen about Cold Fusion Research at BNL.

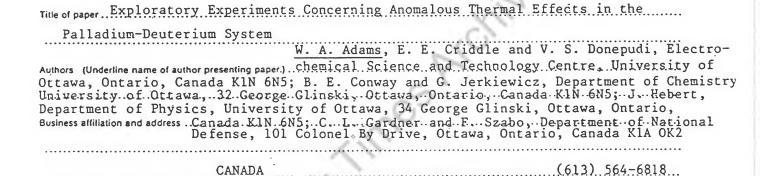
William L. Woodard Panel Secretary

Enclosures



Hollywood, Florida—October 15-20, 1989

This symposium will cover all aspects of cold nuclear fusion. All contributions will be handled as Recent News Papers; hence extended abstracts will not be required. The deadline for submission of 75-word abstracts is September 1, 1989. No paper will be scheduled unless an abstract is received on the standard form by September 1. Abstracts should be submitted directly to the Organizer: Larry R. Faulkner, Dept. of Chemistry, University of Illinois, 1209 W. California St., Urbana, IL 61801. FAX transmission can be arranged by prior telephone contact with L. R. Faulkner at 217-333-8306.



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Electrolysis experiments (in H₂O and D₂O solvent) have been conducted to observe the anomalous thermal and possible cold fusion effects that were recently reported by Fleischmann and Pons (1). Different variables studied include: i) well-characterized palladium electrodes with differing metallurgical structures, ii) electrolyte composition, iii) cell configuration, and iv) the role of recombination. Experimental observations include: i) thermal balance by calorimetry, ii) particle emission, i.e., neutron flux and other possible products, iii) electron-optical analyses of palladium surfaces prior to and following deuterium charging, and iv) analyses of gas streams during deuterium and hydrogen charging.

(1) M. Fleischmann and S. Pons, J. Electroanal. Chem., 261, 301-308 (1989)



Cold Fusion Hollywood, Florida—October 15-20, 1989

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Investigation of Phenomena Relat	ed to D ₂ 0 Electrolysis at	
Palladium Cathode		
	n a t T n n Cohon and	
Authors (Underline name of author presenting paper.)R. Adzic,	D. Gervasio, I. Bae, B. Canan and	
E. Yeager		. .
Business affiliation and address Case Center for Electr	ochemical Sciences, Case Western	
Reserve University, Cleveland, OH		
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(Type abstract in this area-double-spaced.)

Measurements have been performed to check on the Fleischmann and Pons phenomenon. They involved calorimetric measurements in glass cell of the type used by Fleischmann and Pons and also a battery type "Tronac" calorimeter, determination of the D/Pd ratio by coulometry, Li determination in the electrolyte and neutron radiation measurements. Total energy balance of a closed cell obtained by "Tronac" calorimeter showed an excess heat production over an extended period for Pd in LiOD. Much smaller heat effect was found with Pd in LiOH. Heat effect was also found with the glass cell. The D/Pd ratio is 1:1 for 0.25 mm wire. Li was found at 200 nm into Pd bulk. Five-fold enhancement of tritium was found with one cell, while the neutron measurements were inconclusive.



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Cold Fusior Hollywood, Florida—Octob

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Neutrons and Tritium From Cold Fusion in Pd-D
Title of paper
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R. Algasmi*, K. Albertsen, HG. Cnotka and
Authors (Underline name of author presenting paper.) R. Algasmi*, K. Albertsen, HG. Cnotka and
HJ. Schaller
Business affiliation and address Institut für Physikalische Chemie der Universität, Kiel,
F.R. Germany, *The United Arab Emirates University, Chemistry Dept, Alain
United Arab Emirates Post Box #15551 Alain +971-3-634644
(State or Country) (ZIP Code) (Telephone No.)

(Type abstract in this area—double-spaced.)

This report concerns the observation of neutrons and tritium from cold fusion in Pd loaded electrolytically with deuterium. In order to achieve high deuterium concentrations, the electrolysis was carried out at 20, 0, and -80°C, successively using an alcohol acid bath as the electrolyte, Sporadic neutron emissions significantly above the background level were observed in three long time experiments. Measurements of the tritium levels in the electrolyte solutions point to a generation of tritium.

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Cold Fusion

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Title of paper	A Loose Deuteron Cluster Model for (old Nuclear Fusion
	ederline name of author presenting paper.). Dr. Robert T.	Bush and Dr. Robert D. Eagleton
Authors (Un	derline name of author presenting paper.] .	
Business affil	liation and address Physics Department, Califo	rnia State Polytechnic University,
Pomona	3801 West Temple Avenue, Pomona	
	California (State or Country)	

(Type abstract in this area-double-spaced.)

A loose deuteron cluster model (stoichiometries around 1) is presented to attempt to account for cold nuclear fusion. The model accounts for "anomalies" observed in the production of excess heat, neutrons, and tritium.



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	50,		
Title of paper Attempts to Observe Cold Fusion in Pres	sure and Ele	ctrochem	ical
Experiments			
Authors (Underline name of author presenting paper.) M. A. Butler, D. and R. I. Ewing	S. Ginley,	J. E. \$0	hirber.
Business affiliation and address Sandia National Laboratories P.O. Box 5800			
Albuquerque, NM (State or Country)	87185 (ZIP Code)		844-8863 Hephone No.)

(Type abstract in this area—double-spaced.)

The apparent discovery of cold fusion at low levels by Jones and other workers has excited tremendous attention. Neutron production near the background level is difficult to confirm. We report underground experiments with electrochemical cells and pressure bombs simultaneously utilizing three neutron detectors (background 10±1 counts summed over all three detectors). No coincident events have been observed in all three detectors (with appropriate relative intensities) in over 500 hours of run time.



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Title of paper Search for	Evidence of Co	ld Fusion by I	Radiation Dete	ction and Calorimetry
Authors (Underline name of auth	or presenting paper.) E .	Darcy, D. You	ing, G. Badhwa	r, and A. Konradi
Business affiliation and address	NASA-Johnson.S	pace Center/Ei	25, Houston, T	x77.058
	Texas			(713) 483-9055
	(State or Country)	4	(ZIP Code)	(Telephone No.)

(Type abstract in this area—double-spaced.)

Two experiments to verify cold fusion are presented. First, with sensitive radiation detectors, no levels above background were found in vented cells containing 0.1 M ⁶LiOD with three Pd geometries through weeks at 500 mA/cm². Second, precise heat balances are being performed on closed cells with recombination with a high power (20W) heat conduction calorimeter from Hart Scientific, Inc. Cell design and charging sequence are per the proportions and advice of previous work claiming excess heat. Details or apparatus, procedures, and analysis of results will be presented.



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Title of pages	Cold Nucle	ar Fusion	Research	at Cal	Poly Pomona:	Some Pr	eliminary	7
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Data								• • • • • • •
	derline name of author	2	Dr. Robe	rt T. E	ush Dr. Ro	bert D. F	Eagleton 1,	1
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Business affilia	ation and address	1. Physic	s Departm	ent 2.	Chemistry	Departmen	it (Keseai	Cn
Associa	te) - Califo	rnia State	Polytech	nic Uni	versity, Pom	ona, 3801	West	
Temple.	Ave. Pomona,	CA (State or Country)		91768 (ZIP Code)	(714)	869-4019 (Telephone No.)	ł

(Type abstract in this area-double-spaced.)

California state Polytechnic University, Pomona, has begun an experimental effort on cold fusion and preliminary results should be available by conference time. The production of heat and tritium will be checked for a variety of charging currents (above and below 1A) sample masses (1 to 15g), and sample geometries. An attempt will also be made to monitor the emission of neutrons and to detect excess He⁴.



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Title of paper Effect. of Microstructure on the .In	corporation.of.Deu	iterium.into
Palladium		
Authors (Underline name of author presenting paper.)J. W. Flemi	ng, H. H. Law, P.	K. Gallagher,
J. Sapjeta, D. Loiacano, and W. F. Marohn		
Business affiliation and address AT&T Bell Laboratories,	Murray Hill, NJ	
ŊJ		(201) 582-4479
(State of Country)	(ZIP Code)	(Telephone No.)

(Type abstract in this area—double-spaced.)

The effect of microstructure of palladium wires on the incorporation of deuterium was studied by comparing the behavior of annealed and cold-worked samples. Deuterium loading induced greater volume change of the cold-worked samples over the annealed ones. The rate of deuterium loading is also faster in the cold-worked wires.



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Pors (Underline name of author presenting paper.) S. Gottesfeld, R. E. Anderson, D. A. Baker,
C. R. Derouin, F. H. Garzon, C. A. Goulding, M. W. Johnson, E. M. Leonard
T. E. Springer, and T. Zawodzinski
iness affiliation and address
Los Alamos National Laboratory
MEE-11, MS D429
Los Alamos, NM 87545

(State or Country)

(ZiP Code) (Telephone No.)

(Type abstract in this area-double-spaced.)

periments In Search of Muclear Reactions and "Excess Heat" in Metal Deuterides, S. Ittesfeld, R. E. Anderson, D. A. Baker, C. R. Derouin, F. H. Garzon, C. A. Goulding, W. Johnson, E. M. Leonard, T. E. Springer, and T. Zavodzinski. Los Alamos National aboratory

: have investigated claims of nuclear reaction phenomena in low-temperature metal suterides by monitoring Pd-D electrolysis cells and Ti-D high-pressure vessels with igh efficiency ⁵He and NE-213 neutron detectors in a very low background radiation differential heat flux custom-designed calorimetry in 8 lorimeter. Neutron emission was investigated using a multiple detector configuration individual cross-correlation between altiple-detector-correlated events were measured in several weeks of monitoring five fferent Pd-D electrolysis cells or LR2-quenched Ti-D pressure vessels. lorimetric experiments measured the total heat flux from a Pd-D electrolysis cell ntaining a recombination catalyst, which allows for the collection of enthalpic data No energy flux above the input power was observed to date. a closed system.

Please specify audiovisual equipment other than 35 mm slide projector or overhead projector.

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Title of paper A Flow Calorimeter Used in Duplication	on of "Cold Fusion	
Authors (Underline name of author presenting paper.) N. Huang*,	Q.H. Gao*, B.Y.	Liaw*; B.E. Liebert*
Business affiliation and address * Hawaii Natural Energy Inst Engineering, University of Hawaii at Manoa,	itute, ** Depart	ment of Mechanical
Honolulu, Hawaii (State or Country)		808-948-6474 (Telephone No.)

(Type abstract in this area-double-spaced.)

A flow calorimeter was used to measure the excess heat in a Pons-Fleischmann-type "cold-fusion" cell with arc-melted 99.9% Pd as cathode in 0.1M LiOD-D₂O (99.9%) electrolyte. The heat rate was derived from the temperature difference of a steadily circulating water flow between the inlet and outlet of a Dewar flask. Excess heat generation was observed after 30 days of operation.

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Title of paper. Comparison of Thermal Measurements	on Two Fast Mix	ed-Conductor	
Systems: Deuterium and Hydrogen in Pallad	ium		
Authors (Underline name of author presenting paper.) Steven Crouc	h-Baker, Turgut l	M. Gür, George	
Lucier, Martha Schreiber, and Robert A. H	120		
Business affiliation and address Department of Materials S	cience & Engineer	ring	
Stanford University, Stanford, CA	(C)		
California	94305	(415) 723-4110	
(State or Country)	(ZIP Code)	(Telephone No.)	•

(Type abstract in this area—double-spaced.)

Fleischmann and Pons reported that they had observed excess heat generation, neutron and gamma ray emission, and the presence of tritium in electrochemical experiments in which deuterium had been inserted into palladium electrochemically (1). They claimed that this could not be explained by any known chemical or electrochemical effect, and proposed the existence of an hitherto unrecognized form of atomic fusion related to the presence of deuterium inside palladium.

A number of experiments have been undertaken using isoperibolic calorimetry to study the electrolysis of the deuterium-palladium and hydrogen-palladium systems, employing experimental conditions as comparable as possible in order to reduce the possibility of thermal influences due to chemical or metallurgical effects. The results of these measurements, as well as some pertinent experimental considerations involved in the measurement of thermal effects in systems of this type will be presented.

1. M.Fleischmann and S. Pons, J. Electroanal. Chem. 261. 301 (1989)



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Title of paper. Observation of Neutrons from Cold N	uclear Fusion	
Authors (Underline name of author presenting paper.) John N. Harb E. Paul Palmer, J. Bart Zirr, Gary L. Jense		Douglas N. Bennion,
Business affiliation and addressChemical.Engineeringand	Physics.Department	.s.,
Brigham Young University, Provo, UT 846	0.2	
Ut ah (State or Country)		(801) 378-2749 (Telephone No.)

(Type abstract in this area—double-spaced.)

Sensitive neutron detectors were used to investigate cold fusion in two types of electrochemical cells. Low-level bursts of neutrons above background were observed in cells with concentric palladium electrodes. There was no evidence for excess heat or tritium production, although the expected heat generation rates were near the sensitivity limit of the cell. An additional cell capable of very high current densities did not show any measurable evidence of fusion at current densities up to 220 /cm2. Alternate electrode designs and electrolyte compositors have and are being tested, but with no positive results as of this writing. A non-electrochemical system has yielded tentative results of neutron bursts with D₂O prepared Portland cement.



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Title of paper Search For cold Fusion of Deuter	rium.In.Palladium
Authors (Underline name of author presenting paper.) Jacob Jos	rne* and Jan Toke#.
Business affiliation and address *Department of Chemic	al Engineering, #Department of Chemistry
University of Rochester	
Rochester, New York (State or Country)	14627. (716)275-4584. (ZIP Code) (Telephone No.)

(Type abstract in this area-double-spaced.)

Attempts are being made to identify fusion products during the loading of palladium with deuterium. Palladium is being charged with high quantities of deuterium by two methods: (1) Electrolysis of heavy water. (2) Pressurizing palladium in deuterium gas at low temperatures. Effects of current density, temperature, surface and catalytic activities and pretreatment are being studied by positioning neutron counters around the cell to monitor an excessive high energy neutrons and γ -radiation, beyond the background level.

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	f "Electrochemically	Induced Nuclear Fusi	on of Deuterium"-
/ >	t.		
Sow ;	muing paper.) T.R. Jow, F	. Plichta, C. Walker,	S. Slane, and
		chnology and Devices	•
	LABCOM, Power Sources D:	VISION, FOLC HOLLEGELL	
	New Jersey	07703-5000	(201) 544-3630
	(State or Country)	(ZIP Code)	(Telephone No.)

(Type abstract in this area-double-spaced.)

ischmann and Pons¹reported that nuclear radiation and excess heat were erved when deuterium atoms were forced into a cathodically polarized ladium electrode. We have been conducting experiments to attempt to verify caloric claims under similar electrochemical conditions, but utilizing a duction calorimeter rather than the quasi-adiabatic instrumentation of the ginial investigation. The palladium cathodes were wires which were jected to a variety of metallurgical and surface treatments. Control eriments were run using Pd/LiOH, H₂O and Pt/LiOD, D₂O electrodes. Thus far, controls and all other experiments have yielded caloric outputs which are close agreement with simple theory excluding significant "excess" heat.

Fleischmann and S. Pons, Electrochemically Induced Fusion of Deuterium, J. Electroanal. Chem., il, 301 (1989).

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Title of paper Neutron-induced chain-reaction proc	esses for electrol	ysis with metal
deuterides	NI	
Authors (Underline name of author presenting paper.) Yeong F. K	im	
Authors (ordernine frame of author presenting paper.)		
Business affiliation and address Department of Physics		
Purdue University		
West Lafayette, Indiana	47907	(317)494-3042
(State or Country)	(ZIP Code)	(Telephone No.)

(Type abstract in this area-double-spaced.)

Tritium production and excess heat generation observed by Fleischman, Pons, and Hawkins (FPH) and others in electrolysis experiments cannot be explained by known single-step nuclear reactions such as cold deuterium fusions, since the reaction cross-sections and rates are too small at room temperature. However, a combination of known nuclear reactions can form a set of closed chain reactions which can become self-sustaining at a critical stage under favorable conditions and geometries, as in the well-known case of neutron-induced fission chain reactions. The FPH effect is described in terms of chain-reaction processes involving (1) neutron-induced fission-fusion chain reactions and (2) neutron-induced photonuclear chain reactions in palladium deuteride. Experimental evidence and tests of each hypothesis for the FPH effect are described.

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Experiments in Search of Electrochem	rically Induced Cold Fusion
Title of paper	
Authors (Underline name of author presenting paper.)	V.M. Lynes, David Roha,
Reenu Saini and Sarah Rochel-Landau	
Business affiliation and address . Case Western Reserve Univer	sity, Chemical Engineering
Department, A.W. Smith Building, Cleveland,	ОН
Ohio	44106 (216) 368-4132
(State or Country)	(ZIP Code) (Telephone No.)

(Type abstract in this area-double-spaced.)

Calorimeric measurements, tritium balances and neutron counts in electrochemical cells with deuterium charged palladium cathodes are reported.

Control experiments in similar cells but with H₂0-LiOH electrolyte are also provided. Data analysis shows that although a number of the deuterium charged experiments indicate excess power, the signal to noise ratio is too low to unambiguously prove the presence of a non-conventional energy source. A greatly improved experimental system with significantly better accuracy and sensitivity is being designed and will be described.



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Title of paper Calorimetric Studies of Electrochemica	al Incorporat	ion of Hydrogen	• •
Isotopes into Palladium	· 		
Authors (Underline name of author presenting paper.) J. W. Fleming, P. K. Gallagher, and W. Marohn	H. H. Law, J	. Sapjeta,	
Business affiliation and address AT&T. Bell Laboratories. Murra	ay Hill, NJ		
NJ (State or Country)	07974	(201) 582-4479 (Telephone No.)	

(Type abstract in this area—double-spaced.)

The formation of palladium hydride and deuteride by electrolysis has been studied with high precision calorimetry using sealed cells with in-situ recombination of gaseous products as well as open cells. Palladium electrodes prepared by different methods were studied. No unexpected excess heat was observed. The error in the energy balance is estimated to be less than 2%.



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Title of paperDiffusion of Deuterium in Palladium		
Authors (Underline name of author presenting paper.) <u>H.a. H.a. Law</u> ,P.aI	K. Gallagher	. Sapjeta and
Business affiliation and address AT&T. Bell Laboratories Mur	ray Hill, NJ	
NJ (State or Country)	07974 (ZIP Code)	(201) 582-4479

(Type abstract in this area-double-spaced.)

The diffusion of deuterium in palladium foil has been investigated using an electrolytic cell coupled with a mass spectrometer. Diffusion flux is found to be significantly higher when the deuterium content is above 0.4, presumably when the Pd deuteride is essentially all in the beta-phase. A model for the diffusion mechanism is proposed.



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Title of paper. ElectrolysisofLiOD	in.a.Sealed.Cel	1
Authors (Underline name of author presenting paper.)	J. McBreen	
Aumors (Underline name of author presenting paper.)		
Business affiliation and address Department. Laboratory,	of Applied Scien	nceBrookhaven.National
New York (State or Country)	11973	(516) 282-4513 (ZIP Code) (Telephone No.)

(Type abstract in this area—double-spaced.)

The electrolysis of 1 M LiOD was investigated in a cell with a Pd foil (20 mm x 40 mm x 25μ) cathode and a 100 mesh Pt anode. The cell was built like a conventional alkaline electrolyser with an asbestos diaphragm and was run at 1 Amp in a cell containing 120 ml of electrolyte. Sealed operation over a four month period was achieved using a fuel cell electrode to recombine the gases. Measurements included cell voltage, electrolyte temperature and tritium content of the electrolyte. The effect of anions on the entry and egress of hydrogen in Pd was investigated separately. These results will also be presented.



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Calorimetric and Kinet	ic Observat	ion of D ₂ -Pressu	rized LiOD/D ₂ O/Pd
•	,	1/2/	
Cells			
Authors (Underline name of author presenting paper.) . $\stackrel{1}{L}$	Michael C.H.	McKubre, Stuart	I. Smedley,
Francis L. Tanzella and Robert I). Weaver		
Business affiliation and address SRI Internat	ional, 333	Ravenswood Avenu	e, Menlo Park, CA
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(Type abstract in this area—double-spaced.)

Thermal and electrochemical kinetic processes were examined in a cell compromising a Pd cylindrical cathode, concentric Pt coated Ni anode, containing 0.1N LiOD in D_2O . Experiments were performed using an electrochemical overvoltage to load deuterium into the Pd lattice, at a cell voltage below that of electrolysis of D_2O , in an overpressure of D_2 gas.

Results will be described of anomalies in both the thermal behavior evidenced by the calorimetric response, and the kinetic behavior obtained from the interfacial impedance, measured as a function of D_2 loading, as inferred from axial resistance measurements of the Pd electrode.



Special Call for Recent News Papers Cold Fusion Hollywood, Florida—October 15-20, 1989

This symposium will cover all aspects of cold nuclear fusion. All contributions will be handled as Recent News Papers; hence extended abstracts will not be required. The deadline for submission of 75-word abstracts is September 1, 1989. No paper will be scheduled unless an abstract is received on the standard form by September 1. Abstracts should be submitted directly to the Organizer: Larry R. Faulkner, Dept. of Chemistry, University of Illinois, 1209 W. California St., Urbana, IL 61801. FAX transmission can be arranged by prior telephone contact with L. R. Faulkner at 217-333-8306.

Title of paper An Electrochemical Calorimetric Sear	ch For Evidence of	F Cold Fusion
Authors (Underline name of author presenting paper.) M. H. Miles,	K. H. Park and D.	E. Stilwell
Business affiliation and address Chemistry Division, Resear Naval Weapons Center, Chir		
	02555	619-939-1652 (Telephone No.)

(Type abstract in this area—double-spaced.)

Two different types of calorimetric cell designs were used in this attempt to detect excess enthalphy during the electrolysis of $\text{LiOD/D}_2\text{O}$ using Pd cathodes. Control experiments were run side-by-side using water in place of D_2O , Pt cathodes in place of Pd, or by reversing the direction of the cell current. No significant differences were detected between the Pd/D $_2\text{O}$ cells and the controls. For example, in one series of experiments the ratio of heat out to Joule heat in was 1.00 ± 0.04 for one type of calorimetric cell using a Pd cathode in $\text{LiOD/D}_2\text{O}$. For the other type of calorimetric cell, this ratio was 1.065 ± 0.04 in $\text{LiOD/D}_2\text{O}$ compared to 1.075 ± 0.07 in $\text{LiOH/H}_2\text{O}$. Studies on different sources of Pd are in progress.

Please specify audiovisual equipment other than 35 mm slide projector or overhead projector.

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Title of paper Anomalous Heat Production on Electrolyzing Heavy Water Solutions of	
Lithium Deuteroxide with Palladium Cathodes	• • • •
Authors (Underline name of author presenting paper.) A. John Appleby, Young J. Kim, Oliver J. Murphy	?
and Supramaniam Srinivasan	
Business affiliation and address Center for Electrochemical Systems and Hydrogen Research,	
Texas Engineering Experiment Station, Texas A&M University, College Station	2
Texas 77843-3577 (409) 845-8281	
(State or Country) (ZIP Code) (Telephone No.)	

(Type abstract in this area—double-spaced.)

Electrolysis of heavy and light water solutions of lithium and sodium deuteroxide have been carried out for extended periods of time using palladium cathodes of various sizes and geometrical shapes. For palladium cathodes in light water solutions, and platinum cathodes in heavy water solutions, no anomalous heat fluxes were measured using a Tronac microcalorimeter. However, for palladium cathodes in lithium deuteroxide—containing heavy water solutions, of various lithium isotope compositions, anomalous heat fluxes were measured by the calorimeter. Anomalous heat production rates were quenched, or considerably reduced, on exchanging sodium deuteroxide solutions for lithium deuteroxide solutions. Liquid scintillation counting analysis performed on heavy water solutions, after measuring anomalous heat production rates, gave only background levels for tritium in solution. Similarly, analysis for helium isotopes in bulk palladium cathodes (after the evolution of anomalous heat) corresponded to background levels.

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Title of paper Energy Effects During the Electroly:	sis of D ₂ O with Po	l and Pt
Title of paper		
Electrodes	•••••	
Authors (Underline name of author presenting paper.) V.C. Noninsk	i, C.I. Noninski	
Business affiliation and address Laboratory on Electrochemi		nterface
(LEPGER), P.O. Box 9, Sofia 1504, Bulgaria		
Bulgaria		
(State or Country)	(ZIP Code)	(Telephone No.)

(Type abstract in this area—double-spaced.)

Calorimetric experiments are carried out while the electrolysis of D₂O using Pt and Pd electrodes is taking place. It was found out that more energy is being produced than the energy spent for the process. This fact is a confirmation of a similar energy effect, firstly discovered by Fleischmann and Pons. The analysis made of the effect leads to the conclusion that no other explanation can be given for now than the initial proposition made by Fleischmann and Pons that it is due to nuclear fusion.

Please specify audiovisual equipment other than 35 mm slide projector or overhead projector.



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Imposium will cover all aspects of cold nuclear fusion. All contributions will be handled as Recent News Papers; extended abstracts will not be required. The deadline for submission of 75-word abstracts is September 1, No paper will be scheduled unless an abstract is received on the standard form by September 1. Abstracts should mitted directly to the Organizer: Larry R. Faulkner, Dept. of Chemistry, University of Illinois, 1209 W. California bana, IL 61801. FAX transmission can be arranged by prior telephone contact with L. R. Faulkner at 217-333-

paper Calorimetric Measurements of Anomal	ous Power Produc	ed by Cathodic	
Charging of Deuterium into Palladiu	m		
s (Underline name of author presenting paper.) R A Oriani.	J. C. Nelson, S	K. Lee and	
J. H. Broadhu	rst		
ss affiliation and address Department of Chemical Engineering and Materials Science and Dartment of Physics and Astronomy, University of Minnesota, Minneapolis,			
Minnesota	55455	(612) 625-4048	
(State or Country)	(ZIP Code)	(Telephone No.)	

(Type abstract in this area-double-spaced.)

Seebeck-effect calorimetry on the cathodic charging of deuterium into ladium has confirmed that excess power is produced whereas it is not duced with hydrogen. With appropriate experimental parameters the excess wer can be higher than 50 watts per cm³ Pd, and the integrated excess power amount to more than 2.2 MJ/cm³ Pd over eleven hours. Such magnitudes very difficult to rationalize in terms of chemical reactions. At present have no evidence of nuclear reactions.

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Title of paper	ectrochemical and Calorimetric Inv	estigations of the	Pd-D System
The or paper		2	
Authors (Underline n	D. R. Vissers	f. Myles, D. Dees,	M. Krumpelt, and
Business affiliation an	Electrochemical Technology Chemical Technology Divisi Argonne National Laborator	lon	
	9700 South Cass Ave., Arg	gonne, IL 60439-48 (ZIP Code)	37 (312) 972-4518 (Telephone No.)

(Type abstract in this area-double-spaced.)

The purpose of these experiments was to investigate the energetics of the electrochemical formation of Pd-D and Pd-E systems and to find those conditions of cell operations that may produce excess heat useful for energy production. We have performed two series of experiments:

(1) differential comparison of temperature deviations between $(E_2)PdE_1/LiOE$ saturated $E_2O/Pt(O_2)$ and $(D_2)PdD_1/LiOE$ saturated $E_2O/Pt(O_2)$ cells and (2) calorimetric measurements of $(D_2)PdD_1/LiOE$ saturated $E_2O/Pt(O_2)$ cells under varying electrochemical conditions. The E_2O/Pt ratio was monitored during the investigation. Effect of current density and duration of electrolysis on heat generation, value of x, gas recombination, electrode potential, and current efficiency were investigated. No excess heat was observed in long duration experiments within the substantial high sensitivity $(13 \text{ mW/cm}^2 \text{ area of Pd})$ and precision limits of the specially designed constant heat-loss-rate calorimeter.



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	easurements of the H(D) Ti in Pd During Electrolysis	nermodynamic Activi	ty_and
	or presenting paper.)Philip.Ros.	and Harvey Sokol.	
Business affiliation and address .	Materials and Chemical S Lawrence Berkeley Labora		•
		(ZIP Code)	(415) 486-6226

A variation of the classic experiments on Pd charging by Frumkin1 were performed in alkaline electrolytes. Membrane potentials were measured using Pd diffusion tubes having a strain gauge and reference electrode sealed in the "backside" (zero current) of the tube. Frontside (current carrying) and backside potentials and inner hydrogen (deuterium) pressures were measured in 1M KOH(D) and LiOH(D). In KOH, the backside potentials were identical to those reported by Frumkin1 in 1M H2SO4, e.g. n = 42 mV or pzz= 21.5 atm, corresponding to a Pd/H stoichiometry of 0.82 ± 0.02. This activity and stoichiometry was independent of current density, even for current densities as high as 1 A/cm². In KOD, the maximum stoichiometry was slightly lower, 0.78 ± 0.02. In LiOH(D), very different behavior was observed for the frontside potentials, which became very cathodic (n = 1000 mV) at high current density without any corresponding change in the backside potential. Post-mortem analysis of the Pd cathode surface by RBS revealed penetration of Li into the surface region (1-10 µm) during electrolysis at high current density. It is suggested that Fleischmann et al.2 misinterpreted very negative Pd cathode potentials as indicating very high H(D) activity, which our measurements indicate is

A. Frumkin and N. Aladyalova, Acta Physicochim. USSR 19, 1(1944)

² M. Fleischmann, S. Pons and M. Hawkins, J. Electroanal. Chem. 261. 301(1989)

StonyBrook

Department of Chemistry State University of New York at Stony Brook Stony Brook, New York 11794-3400 (516)-632-7905

BITNET: JBIGELEI@SBOOMAIL

FAX: 516-632-7960

MEMORANDUM

Date:

29 August 1989

To:

File

From:

Jacob Bigeleisen

Subject: Cold Fusion Research at BNL

On 17 August 1989 I gave a summary lecture of the Interim Report of the ERAB Cold Fusion Panel at BNL. I was invited to visit one of their research efforts in cold fusion. The work is being done by Harold ("Bud") Wiesmann.

Wiesmann has done calorimetry and is now doing experiments looking for tritium. His calorimetry experiments are not sophisticated. He operates a number of cells, each containing 100 ml. of 0.1 M or 0.5 M LiOD in D_2 0. Temperature is measured with a simple mercury thermometer and the cooling bath is the air flow through the hood, where the cells are located. Thus the bath temperature is good to ± 1°C. His cells therefore operate at significant temperature differentials with respect to the ambient air. He sees no excess heat within about 6%. Even with these experiments it is possible to see that plots of ΔT vs. applied power are not linear. The quadratic term has a negative sign, showing that the heat transfer coefficient increases with temperature.

In all the calorimetric and tritium experiments a set of five cells surround a BF3 counter which is imbedded in a protium rich shield to slow down neutrons. No neutrons above background were detected.

In the first series of tritium measurements 100 ml. of 0.1M or 0.5M solution was reduced to 40 ml by electrolysis. The cathode and anode were platinum wire. This was a control experiment to get a rough value of the electrolytic separation factor. The T content of the $\mathrm{D}_2\mathrm{O}$ increased from 2.58 x $10^{-4} \mu \text{curies/ml}$ (482 dpm/ml.) to 3.74 x $10^{-4} \mu \text{curies/ml}$ (573 dpm/ml.). This corresponds to a separation factor, $k_{\rm D}/k_{\rm T}=\beta$, of 1.68. The temperature is not known, but is above room temperature. This separation factor can be converted to a $k_{\rm H}/k_{\rm D}$ separation factor and is 3.67. The latter is a typical H/D separation factor on platinum cathodes when the hydrogen gas equilibrates with the water. It is a reasonable control experiment, with the caveat that the electrolytic separation factor depends on the type of cathode, inter alia.

The second series of experiments involves a Pd cathode. In these experiments in which six cells were operated, the cells started with 100 ml. of 0.1 M or 0.5M LiOD. There were a total of 150 ml. of D_2 O added during the electrolysis. Thus the volume reduction factor is the same as in the first series, but the enrichment process is different. With each addition there is a dilution of the T that has been enriched in the $D_2^{\,0}$ by electrolysis (or produced by cold fusion) with the stock T in the D20 solution. In this series of experiments the overall separation is 3.43/2.17 - 1.58 compared with 1.45 in the first series using the Pt cathode. The D_0 0 in this series of experiments comes from a different drum at BNL. (2.17 vs. $2.58~\mathrm{x}$ The larger enrichment of tritium in the case of the Pd $10^{-4} \mu \text{curies/ml}$). cathode vs. the Pt cathode is consistent with a larger electrolytic separation factor in the former case. Without additional work it does not provide evidence for the production of a small amount of tritium by a nuclear process within or at the surface of the palladium electrode.

The third series of experiments is currently in progress. experiments the off gas, $D_2 + O_2$ are recombined over a platinum catalyst (not quantitatively) and the tritium activity of the reformed water is The experiments are currently in progress, but definitely show that the $\mathrm{DT/D_{2}(g)}$ is depleted in tritium compared with the starting liquid D20. This confirms the fractionation of T at a palladium cathode during the electrolysis of D₂0. In due course these experiments should yield an independent value for β in the electrolysis of D_2^{0} at a palladium cathode.

Distribution: ERAB panel

H. Wiesmann (BNL)

StonyBrook

Department of Chemistry

State University of New York at Stony Brook
Stony Brook, New York 11794-3400
(516)-632-7905
BITNET: JBIGELEI@SBOMAIL

20 September 1989

FAX: 516-632-7960

Professor Kevin Wolf, Cyclotron Institute, Texas A & M University, College Station, Texas 77843-3366

Dear Kevin,

I am preparing the section on "TRITTUM" for the final ERAB Panel report on cold fusion. I have reports from Bockris and Martin at Texas A & M on their tritium work. I understand that you have also made some tritium measurements on your own. If so, would you kindly complete the attached questionnaire which has been prepared by the panel and circulated by the chairman, Prof. John R. Huizenga.

For those few cases where people have observed tritium I am trying to estimate the secondary neutron production from the well known D + T reaction. For this purpose could you furnish me with the following information concerning your neutron measurements:

- 1. volume of D₂O in cell
- 2. neutron counter efficiency
- 3. counter geometry factor
- 4. neutron counting rate (including those cells that gave tritium and those that did not.

I would appreciate a prompt response. Thank

Jacob Bigeleisen

xc: W. Woodard

TEXAS A&M UNIVERSITY

DEPARTMENT OF CHEMISTRY COLLEGE STATION, TEXAS 77843-3255



September 7, 1989

(409) 845-2011 FAX (409) 845-4719

Professor Jacob Bigeleisen State University of NY at Stony Brook Chemistry Department Stony Brook, NY 11794-3400

Dear Professor Bigeleisen:

Thank you for your recent letter concerning the tritium production observed at palladium during the cathodic evolution of deuterium for D_2O -LiOD.

The concept that some person has added tritium to our samples is a suggestion inconsistent with the widespread observation of tritium under similar circumstances in various laboratories throughout the world (see addendum).

Likewise, your suggestion that the results we have obtained are due to contamination is inconsistent with the exceedingly thorough and insistent investigation of every article which has been utilized in the investigations, including an examination of the glass, rubber tubing and stoppers used in the equipment, etc., and finally with the double testing of the electrodes used for tritium content, both by an electrochemical method here, and by thermal methods at Los Alamos.

The tritium would be present as DT, and in equilibrium with DT in the gas phase. Exchange of DT with water is slow and all T would be readily removed from the liquid, after a burst (It declines ~10x in a few hours (Direct experiment).

As frequent examination for tritium of the systems being electrolyzed over months is not practiced, many investigators may have missed tritium produced in their cells.

I do not understand the relevance of your reference to the work of Farkas, 1937. He measured the H/D separation factor.

We have measured the D/T separation factor, as has also Corrigan at General Motors. It is $2\,\pm\,0.3$.

Under the conditions which we have used (topping up the cells with D_2O on a daily basis so that the total volume remains constant), we calculate (cf. enclosure) that the largest possible increase in T is equal to the separation factor, i.e.,

$$C_{T,t}/C_{T,0} = S - (\$-1)e^{-t/T}$$

I am astounded to learn that you think that the questions concerning the

Professor Bigeleisen September 7, 1989 Page Two

heat production have been substantially clarified. I am in touch with many people obtaining positive results on "cold fusion", and none of them understand the origin of the Heat Bursts. Why do you think they occur?

We are clearly in the first few months of the investigation of the new field of nuclear electrochemistry. I invite you to compare your statement that "bursts of 100 neutrons have no practical implications" to that made by Rutherford at the beginning of the nuclear period in chemistry.

Electrochemical confinement has attained the goal of fusion physics: sustainment. Whether, it will be significant for energy production is an open question decidable in a time inversely proportional to the budget give to research it.

I make two general observations:

1) The phenomena being observed are difficult to investigate because they occur only sporadically and irreproducibly.

The first requirement seems to be very long electrolyses, between 10 and 100 days at high current densities (thus eliminating investigations where the investigators presumably thought they might see something when electrolysis was turned on (maybe 90%); and those in which the current density was less than 100 ma $\rm cm^{-2}$, - most other investigations).

The first goal of future investigations will be to determine why the great time is needed; why various electrodes switch on sporadically, and why the phenomena occur in bursts.

Thus, when a competent investigator such as David Williams at Harwell says he does not observe any phenomena in his system ("but only a few bursts"), I find his statement consistent with the observations in my lab. The difference is one of attitude. For me, the important aspect is the anomalous behavior. A reproduction of the expected result is surely of lesser interest.

2) Scientists in general are wont to eschew the emotional factor of their work.

In fact, the most remarkable aspect of the discovery of electrochemical confinement is the great outburst of negative emotion which has flown from those concerned with rival (but unsuccessful) methods of obtaining confinement. In observing the shaking hand, the red face, the raised voice, the accusations of fraud, or gross stupidity, - I must say that I have never seen anything similar in my long career.

Under such circumstances, in this country, the fledgling phenomena, irreproducible and difficult to find, seem likely to be smothered in howls of rage still being emitted by those who are powerful and who control funding of the needed research.

Professor Bigeleisen September 7, 1989 Page Three

If commercialization becomes scientifically feasible, it seems likely to occur in a nation where minds are less affected by the influence of the greeneyed Goddess. Thus, on August 1, the first of several Japanese Institutes began work - with 80 co-workers.

Yours sincerely,

· Mon. Mun

J. O'M. Bockris Distinguished Professor of Chemistry

JOMB/nr

dections.

ADDENDUM

LABORATORIES OBSERVING PRODUCTION OF TRITIUM DURING THE ELECTROLYSIS OF DEUTERIUM OXIDE AS OF SEPTEMBER 1989:

Packham, Wolf, Velev, Kainthla and Bockris, Texas A&M

Origin: Personal experience and paper

Grozzi, University of Rome

Origin: Personal communication at Santa Fe

Storms, Los Alamos

Origin: Personal visits, very detailed presentation

Ramirez, Institute for Petroleum Research, Mexico City

Origin: Detailed telephone presentation on numerical results to N.J. C.

Packham; paper in Spanish being translated by Maria Gamboa.

Schoessow and Wethington, University of Florida, Gainesville

Origin: Detailed telephone presentation of results

Adzic, Case-Western University, Cleveland Reports 7 x increase in T on telephone.

Iyengar, Bhabha Atomic Research Center

Origin: Paper at Karlsruhe

Landau

Origin: Personal communication and visit to laboratory

Furtak, University of Colorado, reported in Douglas Morrison's News Letter.

Oak Ridge

According to Kevin Wolfe, his August visit to Oak Ridge showed heat in chemistry and tritium in Fusion Physics Departments.

Should you find that any of these statements are not verified, I would be glad if you would let me know.

I note your statement that you must depend upon written statements and not upon rumors. However, it may be that at the present stage, where there is still a great deal of negative feeling among politically powerful fusion physicists, whose work on sustainment has hitherto been unsuccessful, verbal statements are all you may be able to get on the positive side.

Thus, a member of a National Laboratory approached me at a recent meeting and asked me for confidence as to his results. I asked him why I should not pass them on to my colleagues, and he replied with the following immortal words: "My boss would kill me if he knew I was telling you this. You see, they're positive." Correspondingly, a well known electrochemist from a Great Corporation's Lab called me to tell me of 48% excess heat. "I knew I had to get ride of that before my Boss found out".

PERSPECTIVES

Fleischman defends cold fusion

An unperturbed, unshaken Martin Fleischman tried to lay a few ghosts to rest at the IUPAC conference in Stockholm last month. Explaining why he and Stanley Pons had believed that there was a 'marginal' chance of inducing solid state fusion, he cited their that deuterons are theories shielded and exist as D+ in the metal lattice, and further, that D+ behaves as a classical oscillator, resulting in high compression and long confinement times in the lattice. Together these factors could lead to fusion (Chem. Br., 1989, 25, 564). 'We realised', he said, 'that there might be many reasons why this might not work, but we could see some reasons why it might work'.

Referring to the controversial experiments, Fleischman said that the excess heat generated was 109-1012 orders of magnitude above what we had expected from rudimentary measurements of radiation. And after the experiment had been running for hundreds of hours the excess enthalpy was 1-10 MJ cm⁻³ which is 10-1000 times what can be explained by chemistry.

Moreover, while the energy of the cell is increasing the voltage across the cell is decreasing, implying that the energy input of the cell is decreasing-no way can this be explained by chemis-

tryl Fleischman was adamant that he and Pons had done all the control experiments required. 'I think that it is an outrage that we should be accused of such sloppy experimentation', he said. 'Let me assure you that we are more critical of our work than others

Pons and Fleischman are currently concentrating on the excess heat measurements, and have broadened their investigation to include other techniques. They the cheap and cheerful method of heat transfer from a Dewar calorimeter under radiation control; flow calorimetry, which is an absolute technique and does not depend on calibration; and closed calorimetry in which no evolution of gas is allowed, making it a more difficult and potentially more dangerous method.

The latest results coming from the Utah camp indicate that the excess heat is in fact produced in bursts, usually after the experiment has been running for about two months. These bursts of heat can last from hours to days and are of the order of 4 MJ over a couple of days. 'Since we an-nounced these "pulses" at the Electrochemical Society meeting in Los Angeles a couple of months ago (Chem. Br., 1989, 25, 691), several other people have come forward and said that they also have seen such pulses', Fleischman said. But he added that they were unlikely to publish these results because 'they just might be ridiculed as much as we have been'.

So why then have there been so many failed attempts to repeat the cold fusion experiments? Even scientists at Harwell, who ran their experiments continuously for periods of up to six weeks, did not come close to the claims of Pons and Fleischman (Chem. Br., 1989. 25, 769). There are some simple reasons for a lot of the failure', claimed Fleischman. From our work it seems clear that one has to use large electrodes at high current densities for very long periods, and if one is to detect tritium, neutrons or γ -rays we think that the electrode must be polarised for a very long time, and this is difficult'.

Fleischman went further and speculated. 'I think that what we have here is "threshold phenomena"-the main threshold being that it is necessary to reach a certain high current density in order to get the bursts of excess heat that we are observing. We have seen bursts only above 50 mAcm⁻². But clearly at about 500 mAcm⁻² these bursts become more frequent. We are currently trying to develop "heroic electrochemistry"; we are trying to force the system to more extreme conditions to produce the bursts more regularly and continuously. Of course everything falls to pieces when you do that. Incidentally, we have had negative results, and we will of course continue to look for systematic errors to explain them'.

But as anyone will know if they have worked in calorimetry, it is difficult and somewhat unpredictable. Calorimetry on reacting chemical systems is even more difficult-there are many pitfalls and it usually takes several years to develop expertise in it. But as Fleischman said, 'it is important to make mistakes in science—if you only want to be right you will never discover anything'.

Sticking with silicone

Putting an adhesive patch onto your skin seems like an easy way to take medication, but those with sensitive skins will know how easy it is for a sticky patch to become a form of torture. However, the wide range of materials now available means that those allergic rashes could become a thing of the past.

Transdermal drug delivery (TDD)—delivering a drug through drug the skin from one of these sticky patches-has come on in leaps and bounds in the 1980s. Examples of successful TDD include nitroglycerin patches for treating angina pectoris. There are several of these on the market, including Ciba-Geigy's Nitraderm Searle's Nitrodisc and Pharma-Schwarz/Lohmann's Deponit. A new area is progesterone or oestradiol contraceptive patches, such as Ciba-Geigy's Estraderm TTS. Potential also exists for delivanti-inflammatory the NSAID indomethacin locally.

The general pattern of a patch is an impermeable backing layer, which protects the patch from the environment, a matrix or reservoir, which incorporates the drug itself, and an adhesive, which may itself contain the drug. This is covered by a release layer which is peeled off before sticking the patch on.

Skin compatible adhesives so far have included pressure sensitive adhesives (PSAs) based on polyisobutylene and polyacrylate; the newest generation includes silicones. Silicone PSAs were first introduced into medicine in the 1950s and quickly became popular because of their excellent bioility and adhesion.

Silicone PSAs consist of a polymer plus a resin to provide 'tack' the quick stick of initial application. Proper adhesion develops over a period of time (say ca 30 min) after application to the skin. Adhesion depends on the softening temperature of the adhesive—if it is less than the skin temperature (as in silicone PSAs) the adhesive will begin to flow into the folds and creases of the skin.

The low glass transition temperature, T_g , of medical grade silicones is also important because it allows the drug to permeate the adhesive to get to the skin. Low $T_{\rm g}$ silicones show greater conformational flux and rotation of substituents round the silcone Si-O backbone; high $T_{\rm g}$ materials are more 'frozen' and have lower permeability. Clearly this is a parameter that needs to be optimised for TDD.

Medical grade silicone adhesives can also be manipulated by altering the proportions of polymer or resin in the adhesive. Generally they contain a low viscosity dimethylsiloxane polymer with a terminal silanol group -OSiOH). The silicone resin has a three dimensional silicate structure with terminal trimethylsiloxy groups (—OSi[CH₃]₃) and also silanol groups. The adhesive is produced from these component parts by dissolving in a hydrocarbon solvent such as Freon, naphtha or toluene, and in solution the silanol groups crosslink to form SiOSi siloxane bonds. This results in a higher weight polymer, the exact properties of which depend on the degree of crosslinking and the amounts of resin

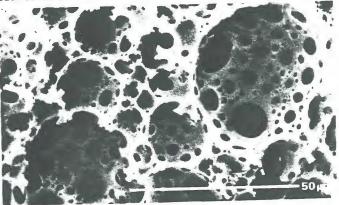
and polymer used. No stabilising additives are needed for silicone PSAs because they are stable over a wide temperature range

More than 85 per cent of today's drugs contain an amino functionality and these groups can initiate crosslinking of silanols and potentially interfere with the PSA properties. The PSA Dow Corning 355, for example, contains a lot of silanol groups and can be so affected, but by trimethylsilylating the silanol groups the crosslinking is inhibited. A new silicone PSA from Dow Corning, BIO-PSA Q7-2920, has been treated in this way to be compatible with most amino-based drugs.

Silicone PSAs are fêted for their adhesive properties so one interesting problem was to find a release layer that did not stick too well to the patch before use! Fluoropolymer layers, with a low permeability and reactivity and low surface activity, have sorted out that one.

Micropores have wide uses

A new range of porous supports for peptide synthesis has been launched this summer by National Starch and Chemical, part of Unilever. The Polyhipe series of supports is based on a rigid porous polymer made by a new emulsion process. The cellular matrix of crosslinked polymer can be used directly or with gels chemically grafted into the structure. Two series have been launched initially: Polyhipe AM is a directly functionalised matrix without gel, while Polyhipe P has polyamide gel bound onto the structure. They can be used in large scale batch or flow peptide synthesis. National is developing a range of products based on this patented emulsion technology, for applications such as cell and enzyme immobilisation and biochemical separations.



'ION

Out of Utah came the promise of the century: cheap, abundant, safe nuclear energy.

But then came the hard questions.







N MARCH 23, DR. B. STANLEY Pons of the University of Utah and Dr. Martin Fleischmann of England's University of Southampton announced that, working together, they had created nuclear fusion at room temperature in a test tube.

Fusion, the process that powers the sun, ordinarily takes place at hundreds of millions of degrees. The announcement that so-called cold fusion had been achieved stunned the scientific community. We, like most of our colleagues, followed the story as it unfolded in the academic and popular press. With a mixture of

excitement and skepticism, we learned that Pons and Fleischmann claimed to be generating four times as much energy as they were putting in, using a small device built with \$100,000 out of their own pockets. Scaled up, the process could conceivably power cities. Small wonder that businessmen and politicians took notice, for such a device would revolutionize the creation and distribution of energy—and with it, the shape of international politics.

"Today we may be poised on the threshold of a new era," declared the chairman of the House Committee on Science, Space and Technology the following month, welcoming Pons and Fleischmann to a Congressional hearing devoted to their achievement. "If so, man will be unshackled from his dependence on finite energy resources."

Alas, by July, the new era had already stumbled and

begun to fall on the threshold.

Today, with their findings almost universally discredited, Pons and Fleischmann continue to cling to their assertion that they have found something new. "We are absolutely sure of our results," Pons told The Wall Street Journal earlier this month. Earlier, Fleischmann had said, "If the amount of heat is so large you can't account for it by chemical reactions, then what else are you going to believe?"

In fact, there may be another factor at work — self-deception.

Pons and Fleischmann apparently fell victim to the experimental scientist's worst nightmare.

Robert P. Crease is an assistant professor of philosophy at the State University of New York at Stony Brook and historian at Brookhaven National Laboratory. N.P. Samios is director of the Brookhaven National Laboratory. Usually, self-deception is quickly and relatively painlessly cleared up, either through the scientist's own labors or those of a neighboring lab bench. What made the case of Pons and Fleischmann different was their meteoric and public rise to celebrity, and their equally spectacular and public downfall.

NUCLEAR ENERGY IS PRODUCED WHEN AN ATOMic nucleus is split, in the process called fission, but it is also produced when two light nuclei are squeezed into one heavier nucleus, a process known as fusion. Fusion, at least theoretically, has several advantages over fission as an energy source. It can be fueled by deuterium, which is easily extracted from ordinary seawater; a fusion reactor would produce less dangerous types of radioactive wastes and would be immune to meltdown. As troubles with fission-generated nuclear power mounted, the promise of fusion power beckoned.

But it was not to be easily achieved. Atomic nuclei are positively charged, and hence repel each other. Bringing two positively charged deuterons — deuterium nuclei made up of a proton and a neutron — close enough to gether for fusion to occur means overcoming an immense electrical repulsion called the Coulomb barrier. The conventional way of overcoming the barrier is to heat up a dense swarm of deuterons and other particles to several hundred million degrees. At those temperatures, the particles move very rapidly, resulting in collisions violent enough to overcome the Coulomb barrier. The scientific and engineering problems involved are enormous, however, and until last spring practical fusion energy seemed to be decades in the future.

Then came Pons and Fleischmann's sensational Salt

Lake City press conference. They claimed, essentially, to have come up with a trick to overcome the barrier. Beginning with a mixture of lithium and heavy water (a form of water made with deuterium rather than ordinary hydrogen), they then inserted two electrodes, a negatively charged electrode made of palladium and a positively charged electrode made of platinum, and passed an electric current through the solution. In their experiment, Pons and Fleischmann found that the temperature of the water rose by an unexpected amount. Calculations showed a fourfold increase in energy output, they said, compared to what they were putting in.

What was causing the increase? Pons and Fleischmann offer the following explanation: As expected, the electric current causes the water molecules to break up and the positively charged deuterons to migrate to the negatively

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Victims of self-deception? Martin Fleischmann, left, and Stanley Pons told Congress they had achieved fusion at room temperature. Below: Turn-of-the-century scientist René Blondiot thought he saw N-rays.



DIANA WALKER/GAMMA-LIAISON



Withelm Roentgen discovered the X-ray by accident, and kept his observations to himself until studies backed him up.

happen. But in science, the miraculous must be regarded with extreme

Eureka! It can



skepticism.

charged cathode, the one made of palladium. Palladium, like other metals, is a crystal, meaning that its atoms are arranged in a regular, tessellated pattern. Its particular atomic structure allows it to absorb deuterons like a sponge. The deuterons lodge themselves in the interstices between the atoms, eventually crowding in two to a space. Powerful electromagnetic forces produced by the palladium atoms squeeze the deuterons together. This pressure, Pons and Fleischmann postulate, pushes the deuterons past the Coulomb barrier and causes them to fuse, with the concomitant release of energy in the form of heat.

Pons and Fleischmann's "trick" was to fuse nuclei not through violent collisions but by compressing them in the miniature vise created by the palladium atoms. No need to create a sun in the laboratory, no need for the equivalent of a hydrogen bomb. Just a tub of heavy water, an electrolyte, two electrodes and some current.

Equally surprising was the relative lack of neutrons produced. Neutrons are a radioactive byproduct of all known fusion (and fission) reactions—and their presence in nuclear reactors is a major factor in making them hazardous places to be. They also contaminate structural elements of the reac-

tor, creating waste-disposal problems. If the excess heat that Pons and Fleischmann reported was due to fusion, then they should have found a neutron flux of 10¹³ neutrons per second in their laboratory — comparable to the amount produced in nuclear reactors. But they reported only 10⁴. They attributed this to the fact that a new form of reaction was taking place. Wonderful! No need for tons of lead shielding, no vexing problem of waste disposal, no need to decommission plants because their parts had become too hot to handle.

At first, excitement mounted as teams of experimenters at the Georgia Institute of Technology, Texas A & M, Stanford University and elsewhere reported apparent confirmations of aspects of the Utah results. Pons, Fleischmann, the University and State of Utah submitted joint patent applications; dozens of corporations contracted to take a look. Palladium futures soared.

Peter Hagelstein, of the Massachusetts Institute of Technology, produced a theory to account for the Pons-Fleischmann observations, and submitted four papers on it to a scientific journal. Utah's Senator Jake Garn proudly chartered a plane for Senate colleagues to fly to Salt Lake City to witness the miracle first-hand. The Utah Legislature offered \$5 million to Pons and Fleischmann for follow-up studies. And the two went to Washington to seek \$25 million for seed money to start a \$100 million Center for Cold Fusion Research.

But it wasn't long before some laboratories reported difficulty duplicating the observations; Georgia Tech withdrew its confirmation. Pons and Fleischmann offered little help to the unfortunates struggling to repeat their work; they declined to provide details of their techniques, refused to send samples of their equipment to laboratories for analysis, and withdrew the paper they had submitted to the scientific journal Nature, claiming that they preferred to press on with more urgent work rather than stop to handle the reviewers' criticisms. Hagelstein's papers remained unpublished; Garn cancelled the charter flight. One by one, laboratories dropped out of the cold-fusion quest, and corporations stopped knocking on Utah's door. In mid-July, a scientific advisory panel to the De-

partment of Energy recommended that the agency not support the work of Pons and Fleischmann. "Evidence for the discovery of a new nuclear process termed cold fusion is not persuasive," the panel concluded. (The D.O.E.'s final report is due in November.)

Scientists began to dub any spurious result a "Utah effect." Wags began peddling "cold-fusion kits," each consisting of a test tube, a pair of electrodes and an Alka-Seltzer tablet, for \$4.95 — "regular price \$5M."

THERE WERE SCIENTISTS WHO HAD SEEN AS EARly as the original press conference that something was amiss, and many more knew after a glance at a pirated copy of the Nature article, which was faxed and refaxed around the world. What tipped them off was the presence of certain tell-tale signs, sometimes known as "symptoms of pathological science." Cold fusion may someday rank among such notorious scientific nondiscoveries as the Martian canals, N-rays and polywater. But it is more than a classic example of pathological science; it also provides a lesson about how experimental science works and why it is enormously difficult.

Self-deception in science is very different from fraud. Few working scientists lose sleep over fraud. The reason is simple: The motive for fraud is almost always résumé-padding — adding lines to a bibliography by publishing fabricated or plagiarized work. But this succeeds only when the work is trivial, insignificant or peripheral; if at all noteworthy, it will be exposed when other researchers try to duplicate and explore it. As Daniel Koshland Jr., editor of the journal Science, put it, "The bigger the result, the more quickly it is going to be checked." A successful fraud may contribute to the culprit's career but it rarely alters the course of science.

Self-deception, however — involuntarily or unconsciously being led astray — is a daily threat to every experimental scientist. Nonscientists often write of researchers as if all they need do is switch on their equipment and — presto! — facts appear, as if the real work of science consists of fitting such facts into theories.

But experimental science is far more complicated—and more interesting—than that. When an experiment begins producing results, the experimenters must still make sure that the readings represent a true profile of a scientific phenomenon—that the data have not been produced by something else in the environment or idiosyncracies of the equipment. To guard against these "systematic effects," as they are called, scientists run experiments over and over, making small changes in the equipment to see whether the effects change. Experimenters typically are suspicious of early data runs, and habitually ask themselves such questions as "How good are these data?" and "What else could they be due to?"

Impure materials, for instance, have ruined experiments. A classic example is the case of a reputable 19th-century chemist named Chenevix. Studying, as it happened, the recently discovered element palladium, Chenevix became convinced that it was not an element at all but a compound of other substances. He circulated his findings among his peers and the general public, ridiculed those scientists who declared it to be an element — and was subsequently horrified to discover that he had erred, apparently by using chemicals that already contained the palladium he believed he was producing from scratch. Utterly humiliated, he abruptly quit science.

Even when the materials are pure, the equipment performs well and is used skillfully, the results must still be "read" correctly.

In the 1930's, a prominent physicist named Irving Langmuir coined the term pathological science, or what he called "the science of things that aren't so." Pathological science, he said, has a characteristic set of symptoms, and he drew up an informal list based on his own experiences. We have drawn up our own, based on ours. It is, we realize, neither exhaustive nor infallible. Genuine science might display any one of these symptoms; pathological science is usually accompanied by several.

Symptom No. 1: Too many miracles.

Miracles do occur in science. On Nov. 8, 1895, the German scientist Wilhelm Roentgen noticed that a fluorescent screen on the table of his laboratory was glowing. Fluorescent crystals glow in the presence of light, but Roentgen

had darkened the laboratory, in order better to examine emissions produced by the cathode ray tube he was using. He switched off the instrument and noticed that the glow stopped immediately. Astonished, and not quite trusting his eyes, he embarked on a systematic study of the phenomenon - fluorescence in the absence of light without mentioning anything to anyone. Finally, on Jan. 1, 1896, he sent a paper to colleagues announcing the discovery of invisible "Xrays," emitted by the cathode ray tube, which were able to pass through paper, wood and even the human body.

Many scoffed; one scientist called the story of the rays a "fairy tale." Every well-stocked laboratory had cathode ray tubes and fluorescent screens, and it seemed highly unlikely that such an effect had not been noticed before. (In fact it had been, but the observations were disregarded.)

Five days later - after scientists had had a chance to read Roentgen's paper - the story of X-rays was splashed across the front page of several newspapers. Within three weeks of the discovery, physicians in Dartmouth, N.H., used them to help them set the fractured arm of a boy named Eddie McCarthy. Roentgen had no "theory" of what the mysterious rays were, hence their name. But when the Nobel Prizes were established in 1901, the first physics award went to him.

Seven years after Roentgen's discovery, a distinguished French scientist, René Blondlot, announced the discovery of "N-rays," which like X-rays were able to pass through paper, wood and sheets of iron, silver and gold. Like Roentgen, he mailed an account of the sensational news to leading scientists all over Europe. N-rays were even more miraculous than X-rays, able to traverse materials in ways that violated known electromagnetic laws. And they were also emitted in some mysterious and unexplained way by living tissue, especially nerves and mus-

Many scientists were disturbed by how much existing scientific knowledge had to be suspended in order to swallow the discovery, and were not surprised when laboratories in Germany and Britain had difficulty reproducing N-rays, or when some laboratories retracted their early confirmations. The rays were exposed as false most dramatically when an American physics professor visiting the lab se-

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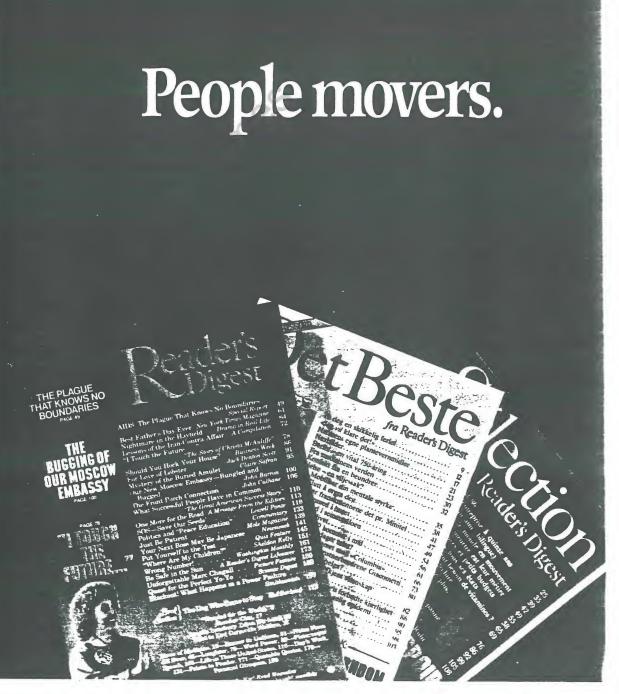
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cretly pocketed a supposedly crucial piece of the apparatus while Blondlot and his assistant continued their observations unperturbed. Blondlot had clearly fallen victim to a grand case of self-deception. The numerous miracles his finding required were an early sign.

Cold fusion, too, required too many miracles. The first was that an utterly unknown way of achieving fusion had escaped the attention of generations of nuclear physicists. The second was that deuterons could be squeezed closely enough together inside palladium for fusion to occur. The third was that the fusion produced so few neutrons. Each miracle, taken separately, was plausible. But the simultaneous appearance of three was strong circumstantial evidence of pathology at work.

Symptom No. 2: The "discoverers" are outsiders.

In the early 1970's, an astrophysicist named McCusker decided to step into particle physics in search of single quarks - fundamental particles with electric charges a fraction of those of the electron and proton. Particle physicists were fairly sure that they came only in trios or in pairs (consisting of a quark and an antiquark) with a total integral charge. Single, isolated quarks had been hunted for years without success. McCusker decided to look anyway - and promptly announced, at a conference in Hungary in 1974, that he had found them.

Again, most scientists first learned of the discovery through the newspapers, and eagerly awaited details. McCusker visited several laboratories, armed cloud-chamber photographs of particles whose tracks were sparser than usual, and asserted that such tracks could only be produced by a particle with a fractional charge.

At Brookhaven National Laboratory, particle physicists asked him whether he had measured the density of each of the tracks in his pictures, whether made by a quark or other particle. He hadn't felt it necessary, he said. When further experiments, replicating McCusker's work, looked at all the tracks, including those of the kind McCusker had ignored, scientists could see that the "quark" tracks had been produced by normal particles and that their sparseness was the result of systematic effects.

McCusker's phantom quarks are but one instance of outsiders making discoveries that turn out to be false. Outsiders start with the disadvantage of hostility from insiders - which they invariably interpret as, at best, the inertial resistance of a hidebound community to bright new ideas, and at worst as xenophobia. But a general skepticism about outsiders is still well-founded. An insider knows the traps, has made the mistakes, and is aware of the temptations. Discoveries are usually made when a person intimately familiar with the field learns or does something and the implications suddenly snap into place. They do happen when someone playing around in unfamiliar terrain stumbles across something that the locals have overlooked - but this is very rare.

Outsiders, however, have the advantage of appearing to the public as Davids in a contest against Goliaths. Pons and Fleischmann, both chemists equipped with the equivalent of slingshots, strode into the province of nuclear physicists, who inhabited huge laboratories well-funded by the Federal government. Add to that romantic image one final touch: the well-known chauvinism physicists tend to have about their field. Small wonder the press cheered, and that editorials in prominent newspapers scorned the "compulsive naysaying" of the establishment's "academic mentality" that dared to question their feat.

As newcomers, Pons and Fleischmann seem to have fallen into several traps that experienced physicists might have avoided. For example, old hands know that where you place the thermometer in a mixture can affect temperature readings. Some physicists have claimed the mixture Pons and Fleischmann worked with should have been stirred to get a uniform temperature; it's unclear whether Pons and Fleischmann did that. Then too, physicists say, Pons and Fleischmann may have encountered difficulties in counting neutrons gamma ravs.

In the long run, scientists paid far more attention to the work of Steven E. Jones, a physicist at Brigham Young University, who at the same time as Pons and Fleischmann's announcement had made a much more modest claim about cold fusion. Jones claims that he detected fusion. but at a level that, as he said, bore the same relation to Pons and Fleischmann's measurements as a dollar bill to the naA scientific paper is like a recipe. If others can't produce the dish, the chef is probably wrong.

tional debt. His colleagues took him seriously not because he was one of their own, nor even because he showed up at all the important meetings to defend his work. Rather, it was because his work betrayed an awareness of potential pitfalls. When Jones reported detecting cold fusion, though at rates far too low to be commercially practical, it was counted as a genuine scientific claim - and

Symptom No. 3: The discoverer has not tried to kill the discovery.

The microbiologist Ludwik Fleck, in "Genesis and Development of a Scientific Fact," a landmark book on the practice of science, compared scientists like Roentgen to Columbus; they set out for an "India" and suddenly run into "America." Inevitably, the discoverer initially is bewildered by the appearance of the unexpected. But most scientists are not as fortunate as Roentgen, and the unexpected turns out not to be a new continent but something produced by faulty or misread equipment. Therefore, the first instinct of a good experimenter, when confronted with the unexpected, is to try to "kill" it - to track down every possible conventional explanation. Anyone prepared to announce the discovery of a new continent should be certain it's not familiar territory misidenti-

A true scientific phenomenon is invariant; it will show up in many different kinds of equipment in different circumstances, somewhat the way a real object can be seen by many different people and from many different angles. Often, an object can momentarily appear to be something it's not - the way, for instance, a photograph might seem to show George Bush with a tourist on a Washington street corner, whereas in reality "Bush" is a cardboard prop. In trying to kill an unexpected result, scientists vary the systematics of the experiment, and make sure to collect plenty of statistics.

Pons and Fleischmann were unable to convince their colleagues that they had made a genuine attempt to kill their results. To all appearances. they had not varied those parameters that one ordinarily varies for studying systematic effects. They hadn't performed the experiment, for instance, with ordinary water instead of heavy water. Nor had they adequately checked the reliability of the neutron detectors. Georgia Tech withdrew its confirmation when it discovered, through systematic checks, that its neutron counters were temperature sensi-

To be sure, the history of science does contain cases of "overkill," in which good scientists have ignored new phenomena staring them in the face. Leon Lederman, former director of Fermilab and winner of the 1988 Nobel Prize in physics, once wrote a paper about "the big ones that got away." In attempting to kill unexpected results by pushing conventional explanations as far as possible, he explained away real breakthroughs, including several that later won their discoverers Nobel prizes. Still, he is in fine company; fellow Nobel laureate Irène Joliot-Curie, to name but one, realized after a rival discovered it that she had observed nuclear fission. But she had convinced herself that the phenomenon could be ascribed to things she already knew.

Symptom No. 4: Inability to repeat the experiment is met by ad hoc excuses. An experimental scientific

paper is in some sense a recipe. It does two things. First, it testifies that the experimenters have a result they want others to know about; second. it provides directions for others to follow. It need not describe every detail - where the bowls were bought, the brand names of the ingredients - but assuming a background set of concepts and a standardized set of laboratory practices, a scientific paper warns, "This is the tricky part. Watch out for that." Scientists establish credibility and priority for discoveries by laying out their work as openly as possible. A scientific paper with an inadequate recipe is a tip-off that the authors' understanding of their work is incomplete.

Blondlot, for instance, was never able to provide a sufficient recipe for N-rays. When his colleagues said that they couldn't detect the rays, Blondlot produced additional details, accompanied by dark

suggestions that those couldn't repeat his work incompetent. A few of B lot's champions even b claiming that only indials of Latin descent had enough eyesight to see dence of the rays.

Pons and Fleischmann lowed this pattern. V someone claimed that it not possible to produce fusion, the two Utah s tists would add more insi tions - that the pallac cathode had to be prepara certain way, or the elelyte had to be of a certain centration. As Robert F head of the Washington o of the American Phy Society, remarked, time someone did the ex ment with no result; would say, 'You didn't de experiment right,' and up another tidbit."

HY DOES PA ological sci occur? The r vation may be a desire fame, immortality, weal or a genuine desire to mankind. Fleischmann stated that the motiva for the Utah team's v was social: "We recog the desperate energy n that will confront the century," he said. But if desires interfere with (disinterested evaluation testing, the outcome ma pathological science:

If Pons and Fleischman tually achieved what claimed, to get the scier community to change its about cold fusion they have to start over scratch - by publishing is scientific literature, de strating expertise in the p dures, showing that they tried and failed to kill thei sults, and providing a con hensive set of instructions others can follow.

In the movie "Back to Future" the time-trave DeLorean is powered t small device that runs beer. The label reads:

Mr. Fusion

Home Energy Reactor The scene effectively tures, and sends up, the science seems to be stantly discovering, simp ing, standardizing and m turizing complex and poful phenomena in such a that they can be handle ordinary people with scientific training at all. world may someday cor fusion devices that will t convenient as automatic fee makers. But if so, ti will be no shortcuts in scientific process lea from here to there.

Dallo Utah: scusateci, la fusione fredda è un sogno

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Oggi Replay duplica

<u>In palio</u> 20 milioni

Correte a pagina 5

SALT LAKE CITY — Un'altra doccia gelata sulla fusione fredda. Anche all'università dello Utah, dove gli scienziati Pons e Fleischmann diedero il grande annuncio nel marzo scorso, sono costretti ad ammettere che tutti i tentativi fatti finora per riprodurre la fusione nucleare a freddo sono falliti.

Non è escluso che presto dovrà chiudere i battenti il neonato Istituto nazionale della fusione fredda, inaugurato grazie ai sei miliardi di fondi forniti prontamente dal governo.

Nessuna delle «pile» elettrochimiche (più di venti) predisposte dall'I-stituto per duplicare l'esperimento che i due chimici affermarono di avere eseguito più volte in barattoli di vetro a temperatura ambiente ha dato energia maggiore di quella immessa o prodotti tipici della fusione nucleare. Pons e Fleischmann, in questi giorni, sono in Giappone per alcune conferenze.

A pagina 7

OGG]

CORRIER CONOMIA

Cresci America cresci Un'inchiesta sulla Bushnomics

La lira? E' forte Sondaggio fra i banchieri internazionali

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Moda
I giapponesi a caccia di
griffe italiane

Immobili
De Benedetti conquista
piazza Scala

Perestrojka padana Il sogno bolognese Oggi con il Corriere



La seconda guerra mondiale

raccontata da ENZO BIAGI

Nel secondo fascicolo: I tedeschi a Parigi -Olanda e Belgio a ferro e fuoco - In Norvegia la guerra tra i fiordi - Dunkerque: la vittoriosa sconfitta -Compiègne: il vagone della vendetta.

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posta boundaria con una lievissima p

cialista»,

Sono falliti tutti i tentativi di riprodurre il controverso esperimento di Pons e Fleischmann

Dagli USA doccia gelata sulla fusione fredda

SALT LAKE CITY — Un'altra doccia gelata sulla fusione fredda. Anche all'università dell'Utah, dove gli scienziati Pons e Fleischmann diedero il grande annuncio a marzo, sono costretti ad ammettere che tutti i tentativi fatti finora per riprodurre la fusione fredda sono falliti e non si esclude di dover presto chiudere i battenti del neonato Istituto nazionale della fusione fredda, inaugurato grazie ai sei miliardi di fondi forniti prontamente dal governo.

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Nessuna delle «pile» elettrochimiche (più di venti) predisposte dall'Istituto per duplicare l'esperimento che i due chimici affermarono di avere eseguito più volte in barattoli di vetro a temperatura ambiente ha dato energia maggiore di quella immessa o prodotti tipici della fusione nucleare, cioé neutroni e trizio.

«E' arrivato il momento di incominciare a chiederci se facciamo qualcosa di sbagliato», ha dichiarato Huro Rossi, direttore dell'Istituto. E' una situazione «snervante», ha Pons e Fleischmann si trovano attualmente in Giappone e il loro ritorno non è previsto prima di giovedì, per cui non si conosce ancora la loro reazione. Le dichiarazioni fatte da Rossi danno ragione allo scetticismo con il quale il mondo scientifico internazionale accolse l'annuncio dei due chimici, che il 23 marzo prospettarono la possibilità di avere a portata di mano una fonte inesauribile di energia pulita e a buon mercato.

Secondo Pons, preside della facoltà di chimica dell'università dell'Utah, e Fleischmann, inglese, dell'università di Southampton, con il loro metodo sarebbe possibile con un secchio d'acqua di mare ottenere energia pari a quella fornita da una tonnellata di carbone.

I due sostennero che con un processo elettrochimico attuato dentro un vaso di vetro grande come un barattolo da cucina era possibile spingere due deuteroni (particelle nucleari di deuterio o idrogeno pesante, che abbonda nell'acqua di mare) attraverso un reticolo formato da elettrodi di palladio e platino, otte-

nendo la fusione di particelle con carica positiva e creando così nuovi atomi.

«Le indicazioni che abbiamo tratto dalle nostre ricerche ci dicono che la scoperta verrà con relativa facilità trasformata in una tecnologia utilizzabile per generare calore e energia», disse all'epoca Pons. Nonostante le critiche subito piovute da tutto il mondo su i due chimici, l'università dell'Utah brevettò l'esperimento Pons-Fleischmann e il governo dello Stato trovò senza indugi 4,3 milioni di dollari per creare «The national cold fusion institute».

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Le dichiarazioni di Rossi sono giunte dopo la visita compiuta all'università la settimana scorsa da un esperto del Cern, il laboratorio europeo per la ricerca nucleare con sede a Ginevra. Si tratta di Douglas Morrison, che ebbe una serie di incontri con gli scienziati di Salt Lake City e dichiarò poi che i tentativi eseguiti in tutto il mondo per duplicare la «fusione fredda» lasciano ben poche speranze che sia fattibile.

PRBIT SCRIPT Q1 dated 89/09/28 08:28:26 Page 1

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Wed, 27 Sep 89 19:48:06 PDT Date:

From: hoffman@Csa2.LBL.Gov (Darleane Hoffman) Message-Id: <890927194806.21c0238d@Csa2.LBL.Gov>

Subject: Re: Slightly revised first draft

In-Reply-To: Your message <890924193329.20800170@Csa2.LBL.Gov> dated 24-Sep-1989

schiffer%anlphy.bitnet@Csa2.LBL.Gov To:

jbigelei%sbccmail.bitnet@Csa2.LBL.Gov, rlg2@ibm.com, cc:

koonin%caltech@Csa2.LBL.Gov, hoffman@Csa2.LBL.Gov

Dear John,

I have received your slightly revised draft. I would prefer to amend my section on protons as per the FAX I sent you. I will send that via electronic mail so it will be easier for you to incorporate in the draft. Did you get my message concerning the correction of Porter et al.? Please confirm. Also I have checked with Buford di ,ur won Price and his paper has been submitted to Nature and we can distribute it as widely as we wish so I will FAX a copy to each of our working group members.

Date: Wed, 27 Sep 89 14:49 EDT From: JBIGELEISEN@SBCCMAIL.BITNET

Subject: NSF - EPRI Workshop on Cold Fusion

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State University of New York at Stony Brook Stony Brook, NY 11794-3400

Jacob Bigeleisen
Professor
Chemistry
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27-Sep-1989 02:33pm EDT

FROM: JBIGELEISEN

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CC: Remote Addressee (_RLG2@YKTVMV.BITNET)
CC: Remote Addressee (_KOONIN@CALTECH.BITNET)
CC: Remote Addressee (_HOFFMAN@LBL.GOV)
CC: Remote Addressee (_SCHIFFER@ANLPHY.BITNET)

SUBJECT: NSF - EPRI Workshop on Cold Fusion

John,

I was at BNL this morning and saw Peter Bond, Chairman of Physics and BNL coordinator for "cold fusion". He told me that NSF and EPRI were jointly sponsoring a workshop on cold fusion sometime in October. I believe it may be about the time of our Chicago meeting. Apparently the only people being invited are those who sho "positive" results. I believe that DOE should take this matter up with NSF. As a minimum, we should send Dave Goodwin and some additional DOE staff personnel as observers - you know the Freedom of Information Act. I took the opportunity to tell Peter Bond that I was going to recommend to DOE that he have his budget cut. During the last month the BNL cold fusion people have had the following outside speakers that I know of:

- 1. Srinivasan Texas A&M
- 2. Huggins Stanford
- 3. Jones BYU
- 4. Inyengar of BARC is coming on 9 October.

Inyengar's visit is the first one I have heard about before the fact. I will not go to it. I will be in the Adirondacks (much better use of time).

Through the efforts and interests of Maurice Goldhaber and Joe Weneser I gave a seminar talk at BNL on the findings of our panel in mid August. There are some funny things goind on at BNL with respect to their cold fusion effort, if that is what one can call what they are doing.

JAKE

Dallo Utah: scusateci, la fusione fredda è un sogno

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Oggi Replay duplica

<u>In palio</u> 20 milioni

Correte a pagina 5

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Non è escluso che presto dovrà chiudere i battenti il neonato Istituto nazionale della fusione fredda, inaugurato grazie ai sei miliardi di fondi forniti prontamente dal governo.

Nessuna delle «pile» elettrochimiche (più di venti) predisposte dall'I-stituto per duplicare l'esperimento che i due chimici affermarono di avere eseguito più volte in barattoli di vetro a temperatura ambiente ha dato energia maggiore di quella immessa o prodotti tipici della fusione nucleare. Pons e Fleischmann, in questi giorni, sono in Giappone per alcune conferenze.

A pagina 7

OGGI

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Cresci America cresci Un'inchiesta sulla Bushnomics

La lira? E' forte Sondaggio fra i banchieri internazionali

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Sirene italiane a Washington

Ventriglia
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raccontata da ENZO BIAGI

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«E' arrivato il momento di incominciare a chiederci se facciamo qualcosa di sbagliato», ha dichiarato Huro Rossi, direttore dell'Istituto. E' una situazione «snervante», ha detto. Pons e Fleischmann si trovano attualmente in Giappone e il loro ritorno non è previsto prima di giovedì, per cui non si conosce ancora la loro reazione. Le dichiarazioni fatte da Rossi danno ragione allo scetticismo con il quale il mondo scientifico internazionale accolse l'annuncio dei due chimici, che il 23 marzo prospettarono la possibilità di avere a portata di mano una fonte inesauribile di energia pulita e a buon mercato.

Secondo Pons, preside della facoltà di chimica dell'università dell'Utah, e Fleischmann, inglese, dell'università di Southampton, con il loro metodo sarebbe possibile con un secchio d'acqua di mare ottenere energia pari a quella fornita da una tonnellata di carbone.

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«Le indicazioni che abbiamo tratto dalle nostre ricerche ci dicono che la scoperta verrà con relativa facilità trasformata in una tecnologia utilizzabile per generare calore e energia», disse all'epoca Pons. Nonostante le critiche subito piovute da tutto il mondo su i due chimici, l'università dell'Utah brevettò l'esperimento Pons-Fleischmann e il governo dello Stato trovò senza indugi 4,3 milioni di dollari per creare «The national cold fusion institute».

6

Le dichiarazioni di Rossi sono giunte dopo la visita compiuta all'università la settimana scorsa da un esperto del Cern, il laboratorio europeo per la ricerca nucleare con sede a Ginevra. Si tratta di Douglas Morrison, che ebbe una serie di incontri con gli scienziati di Salt Lake City e dichiarò poi che i tentativi eseguiti in tutto il mondo per duplicare la «fusione fredda» lasciano ben poche speranze che sia fattibile.



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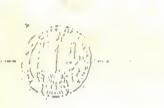
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FAX TRANSMITTAL ****** Richard Garwin IBM Fellow & Science Advisor to the Director of Re:earch TBM CORPORATION Box 218 YORKTOWN HEIGHTS 10598 914-945-2141 FAX NUMBER: Law ence Barkeley La Professor Darleane C. Hoffman Be 'ke' ey, CA 94720 70A/3307 September 20, 1989

Search for MeV Ions Produced during Fracture of LiD Crystals P. B. Price

Physics Department, University of California, Berkeley, CA 94720

Recent experiments have shown 1-5 that the rate of cold fusion of deuterons curing either electrolysis or high-pressure deuteration of Pd or Ti is many orders of magnitule lower than claimed⁶⁻⁸. It has been conjectured^{9,10} (Mayer, F. J., King, J. S., and Rei z, J. R., unpublished; and Ryan, R. et al., unpublished) that fusion, which would occur at vanishingly low rates under static conditions, might occur at detectable rates under dy nar tic conditions. In particular, if a metal lattice, embrittled by deuteration, were to under go microfracturing, deuterons might be accelerated in the transient high electric fields across the cracks, reaching energies sufficient to cause fusion. Emission of low-energy io as 1 as been observed during mechanical fracture of TiDo.8 (Menlove, H. O. et al., unpublishe i), and Klyuev et al.11 have claimed that, during fracture of LiD single crystals, they detec ed an excess of neutrons above the cosmic-ray background. These results, and earlier clai ns to have observed electrification 12,13 and emission of low-energy electrons 14,15 and ion;15 during fracture of dielectric crystals, prompted me to see whether energetic protons are emitted during fracture of LiD crystals. My resulting null result casts doubt both on the claim of Klyuev et al. to have seen fusion neutrons and on the conjecture that deuterated Pd or Ti could produce a detectable number of energetic particles as a result of fusion at стаскs.

Figure 1 shows the geometry of the experiment. I obtained a ~15 cm³ single crystal of LiD (grown by M. Delong, Physics Department, University of Utah). I cleared the crystal vertically along (100) planes into about 10² thin plates, with the bottom of the crystal supported 0.9 mm above the surface of a CR-39 track-recording plastic detector. This distance is negligible compared with the range in air of either a 3.03 MeV proton or a

1.01 MeV triton emitted in the fusion reaction $d+d \rightarrow p+t$. To eliminate background tracks due to radon decay and cosmic-ray interactions, I etched the CR-39 and located pre-existing tracks just before cleaving the LiD crystal. In this way, as in a previous search for cold fusion in my laboratory⁵, the background was reduced to zero. After cleaving the crystal 10^2 times, I re-etched the CR-39 and scanned it for new tracks. There were 9 pre-existing tracks of particles with Z=1 and 2, and no new tracks. The etching conditions were the same as in ref. 5.

Table 1 compares my experiment with that of Klyuev et al. ¹¹ Each fragment of the single crystal of LiD that I cleaved was ~2.5 cm long and 0.4 to 2.5 cm high. However, the ranges of a 3.03 MeV proton and of a 1.01 MeV triton in LiD are only ~0.19 mm and ~0.015 mm, respectively, much shorter than the height of the cleavage crack. Thus, the tritons sample only ~8% as large an area of crack as do protons and can be ignored. We have to take into account not only the solid angle and efficiency for detection but also the surface area of crack from which protons could reach the detector and be recorded. Assuming that fusion products are emitted isotropically and uniformly throughout the cleaved samples, the product of detection efficiency, η , and effective source area, A, is $<\eta A>=0.0057$ cm² for protons in my experiment, compared with 0.0023 cm² for neutrons in the experiment of Klyuev et al. (In my experiment the cracks ran the full horizontal length of each crystal, ~2.5 cm; the effective height was ~0.019 cm; and the maximum acceptable zenith angle was ~40°; see ref. 5 for a more detailed discussion of the calculation of $<\eta A>$.)

Based on the average number of neutrons per fracture reported by Klyuev et: 1., and taking account of the fact that the production rates of 3 MeV protons and 2.45 M:V neutrons in d-d fusion are about equal, I expected to detect ~64±27 protons per 100 cleavages, whereas I found none. At 90% confidence level, the null result of my search for protons allows one to infer that the number of neutrons per fracture of an area 0.09 c n²

should be less than ~10-2. If the number of neutrons produced scales with crack area, he upper limit (90% C.L.) becomes one neutron per 9 cm².

We can use this limit to test the conjecture that d-d fusion might occur in he transient electric fields across a propagating crack. Let us make the rather extre ne assumption, that deuterated Pd or Ti undergoes brittle fracture as readily as does LiD and develops as large an electric field across a crack as does LiD. (The latter seems highly unlikely, since LiD has a remarkably low conductivity at room temperature, ~10-23/oh n-cm.) The upper limit for deuterated Pd or Ti then becomes one neutron generated by tusi on for every 9 cm² of crack area formed. To account for even the lowest claimed cold tusi on yield, ~10³ neutrons cm⁻³ during an 18 hr run (Menlove, H. O., et al., unpublished), at least 10⁴ cm² of crack area would have to be generated. These authors claimed that the neutrons often occurred in bursts of up to 250 neutrons, which would require the creation of more than 2500 cm² of crack area at one time. This stretches credulity. To attribute the origin of the vastly higher neutron fluences in the original reports of cold fusion to a crack mechanism would require the creation of ~10¹⁰ cm² (ref. 6) and ~10⁵ cm² (ref. 7) of crack area.

My conclusions are that (a) positive ions such as are detected during fracture of a wide variety of materials are not accelerated to MeV energies; (b) the number of neutro is emitted per cm² of crack area in LiD is at least a factor 30 lower than reported in ref. 11; (c) fusion of deuterons accelerated in the electric field across a crack in a deuterated material cannot account for any measurable neutron fluences.

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ACKNOWLEDGEMENTS I thank Michael Solarz for experimental assistance. The work was supported in part by the National Science Foundation.

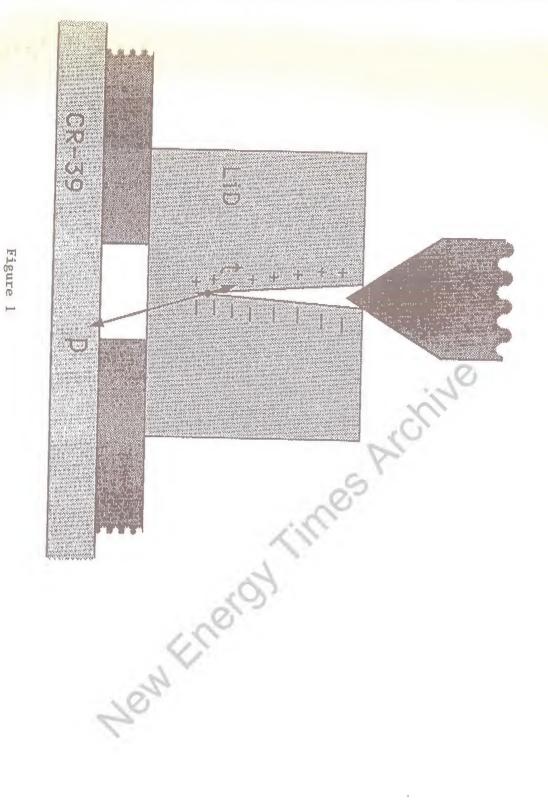
Table 1 Comparison of this experiment with that of Klyuev et al.

	Klyuev et al.11	This expt.
Effective source area per cleavage	~0.09 cm ²	~0.04 cm ²
Detection efficiency (incl. solid angle)	2.6%	12%
Number of cleavages	several	~102
Particle studied	neutrons	protons & tritens
Observed number of particles	0.26±0.11 per fracture	0
No. expected, based on Klyuev et al.	erc ep	54 ± 23
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Figure Caption

1. System for recording tracks of protons and tritons from d-d fusion resulting from acceleration of deuterons in the transient electric field across a cleavage crack.

New Fines Archive



Received: from Csa2.LBL.Gov by IBM.COM (IBM VM SMTP R1.2.1MX) with TCP; Wed, 27 Sep 89

22:34:00 PDT

Date: Wed, 27 Sep 89 22:32:16 PDT

From: hoffman@Csa2.LBL.Gov (Darleane Hoffman)
Message-Id: <890927223216.21c02480@Csa2.LBL.Gov>

Subject: Re: Slightly revised first draft

In-Reply-To: Your message <890924193329.20800170@Csa2.LBL.Gov> dated 24-Sep-1989

To: schiffer%anlphy.bitnet@Csa2.LBL.Gov

cc: jbigelei%sbccmail.bitnet@Csa2.LBL.Gov, rlg2@ibm.com, koonin%caltech@Csa2.LBL.Gov, hoffman@Csa2.LBL.Gov

To: John Schiffer From: Darleane Hoffman

Corrections to Table II.

The yield normalized to Jones et al. for Sundqvist et al. should be 0.2 not 2. Also note spelling of Sundqvist.

There should not be an [a] after Porter et al. as they state that they did correct for the geometric collection efficiency and the limited volume of the Pd foil which was sampled using the same method as Price et al. [Pr].

Please replace the first part of my section IV. with the following:

experiments (Po, Pr, Re, Su) to measure the 3 MeV protons and/or the 1 MeV tritons produced in the reaction, D(d,p)T, have been reported; they are summarized in Table II below. A variety of different methods has been used, but the lowest limit on chargedparticle production appears to be that set by Price et al. (Pr) using plastic track detectors. Their setup was designed so that the light water control cell matched the heavy water cell as closely as possible. Cathodes of 25-micrometer-thick Pd sheet with a 100 nm Au diffusion barrier evaporated on the side which faced the plastic (CR-39) detector, and anodes of 2 micrometer Pd evaporated on 100 nm Au on microscope slides were used. The light water control cell contained 1 M LiOH and H2O and the other cell contained 1 M LiOD and D2O. Electrolysis was performed for 13 days, and the cathode stoichiometry determined to be Pd(H,D)0.8. Both cells showed track production rates which agreed and were consistent with the alpha-particle rate for native Pd foils due to trace (ppm) impurities of the natural U-238 and Th-232 decay chains; however, no tracks due to protons with energies between 0.2 and 3 MeV or tritons with energies between 0.2 and 1 MeV were found. From these data Price et al. (Pr) set limits on the fusion rate of less than 0.002 per cm3 This value results in an upper limit of $8.3 \times 10-26$ second. fusions per dd pair per second. This is about an order of magnitude lower than the limits obtained using Si surface barrier (SSB) techniques.

A limit on the fusion rate of 0.0281 per cm3 per second or 1.2 x 10-24 fusions per dd pair per second was obtained by Zeigler et al. [Zi] using a SSB technique. Porter et al. [Po] used a SSB detector to view the back of a 76 micron thick Pd foil cathode in a heavy water electrolysis cell. They obtained a limit of less than 6 x 10-25 protons per dd pair per sec at the 2 sigma level; chemical analysis of their electrolytes showed no evidence for anomalous increases in tritium concentrations. Sundqvist et al. [Su] also used a SSB technique to detect protons. The detector was placed close to Pd foil cathodes which were thin enough to allow all the protons produced to escape from the foil. All of their runs gave a result of 0 within the statistical errors, resulting in a fusion rate of

(-2.1+/-2.2) x 10-24, if a bulk process is assumed.

Recently, Rehm et al. Re have reported using a proportional counter to search for charged particles from electrolytic cells with Pd and Pt electrodes in 0.1 M LiOD in D20. They obtained an upper limit of 4 x 10-23 fusions per dd pair per second, not as low as the limits using the other methods.

In summary, a variety of experimental techniques has been used in searches for charged particles; all of them set very low limits on fusion occurring via the D(d,p)T channel. Most of these results set limits on fusion via this channel that are considerably less than Jones et al.'s [Jo] value of (1.00+/-0.82)x10-23 fusions per dd pair per second for the D(d,n)3He channel obtained from neutron (Sundqvist et al. calculated the uncertainty for this measurements. value.)

The upper limit of Price et al. [Pri of 8 x 10-26 fusions per dd pair per second is much below the average low rate inferred from the neutron measurements of Jones et al.5 or even those of Menlove et al. Me. The extremely low limits which the searches for charged particles (either protons or tritons) place on their production is clearly inconsistent with the reported production of tritium via the cold fusion reaction."

I would also like to add something referring to the recent work of Price [Pr2], which has been submitted to Nature, probably right after the above which says:

"Very recently, Price [Pr2] has cleaved a 15 cc single crystal of LiD vertically along (100) planes into about 100 thin plates, with the bottom of the crystal supported 0.9 mm above the surface of a track-recording plastic detector. Based on the average number of neutrons per fracture reported by Klyuev et al. [Kl], and the nearly equal production rates of 3 MeV protons and 2.45 MeV neutrons in DD fusion reactions, he expected to detect about 64+ -27 protons per 100 cleavages whereas none were found. Assuming the number of neutrons produced scales with crack area, he set an upper limit at the 90% confidence level of one neutron per 9 cm2. In order to account for even the lowest claimed cold fusion yield of about 10exp3 neutrons per cc, at least 10exp4 of crack area would be required. If the much higher neutron fluences reported originally [F1], [Jo] for cold fusion were to be attributed to a crack mechanism, creation of crack areas of the order of 10exp10 and 10exp5 cm2, respectively, would be required! He concludes "that positive ions such as are detected during fracture of a wide variety of materials are not accelerated to MeV energies; that the number of neutrons emitted per cm2 of crack area in LiD is at least a factor of 30 lower than reported by Klyuev et al. [K1]; and that fusion of deuterons accelerated in the electric field across a crack in a deuterated material cannot account for any measurable neutron fluences"."

REFERENCES

- P. B. Price, S. W. Barwick, W. T. Williams, and J. D. Porter, "Search for Energetic Charged Particle Emission from Deuterated Ti and Pd Foils", Phys. Rev. Lett., submitted 7-14-89
 [Pr2] P. B. Price, "Search for MeV Ions Produced during Fracture of
- LiD Cyrstals, September 1989 Preprint, Nature (submitted).
- Zil J. F. Ziegler, T. H. Zabel, J. J. Cuomo, V. A. Brusic, G. S. Cargill III, E. J. O'Sullivan, and D. A. Marwick, Phys. Rev. Lett. 62, 2929 (1989).
- [Po] J. D. Porter, A. A. Shihab-Eldin, H. Bossy, F. J. Echegaray, J. M. Nitschke, S. G. Prussin, J. O. Rasmussen, and M. A. Stoyer, "Limits on Electromagnetic and Particle Emission for Palladium-D2O Electrolyt-

ic Cells", Lawrence Berkeley Laboratory Preprint LBL-27523, July 1989, submitted to J. Fusion Technology.

[Sul B. U. R. Sundqvist, P. Hkansson, A. Hedin, R. V. Bucur, B. Johansson, and R. Wppling, Uppsala University, Sweden, preprint of o9o5 manuscript accepted for publication in Physica Scripta, May 15, 1989.

[Jol S. E. Jones, E. P. Palmer, J. B. Czirr, D. L. Decker, G. L. Jensen, J. M. Thorne, S. F. Taylor and J. Rafelski, Nature 338, 737 (1989).

Me H. O. Menlove, M. M. Fowler, E. Garcia, A. Mayer, M. C. Miller, R. R. Ryan, and S. E. Jones, "Measurement of Neutron Emission from Ti and Pd in Pressurized D2 Gas and D2O Electrolysis Cells", Los Alamos National Laboratory Report LA-UR 89-1974, submitted to Nature, June, 1989.

As to the quotation, I am very fond of it, but our Committee has already been accused of being biased against cold fusion and I'm afraid this will make us appear snide as well--although perhaps that doesn't really matter!

More comments later as soon as I have a chance to read more carefully.

Darleane

PRBIT SCRIPT Q1 dated 89/09/27 18:14:44 Page 1

Date: Tue, 26 Sep 89 13:00 EDT From: JBIGELEISEN@SBCCMAIL.BITNET

Subject: Comments on Schiffer Draft of 24 Sept.

To: RLG2@YKTVMV.BITNET

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> Jacob Bigeleisen Professor Chemistry 516-632-7905 26-Sep-1989 12:57pm EDT

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SUBJECT: Comments on Schiffer Draft of 24 Sept.

26 September 1989

Dr. John P. Schiffer, Physics Division, Building 203, Argonne National Laboratory, Argonne, Illinois 60439-4843

Dear John,

It is clear why John Huizenga asked you to coordinate the section on fusion products. It is excellent; I have afew comments. I will not try to deal with the typographical errors.

1. I believe we need to cover Menlove's neutron bursts both from metals in contact with D2 and from his electrolytic cells. This is important not only as one of a number of experiments that give neutrons at low levels, but also it points out the difficulties in detecting low levels of neutrons. This has implications for other programs for which Menlove built these detectors.

2. We need a section on 3He and 4He. Measurements have been made at Livermore, Battelle Northwest and North American Rockwell. Fleischmann and Pons have sent heat producing electrodes to Johnson and Mathey some time ago. There is no word of their findings. The other analysts report less than 10**9 He atoms in the samples provided to them. From the heat burst

and excess heat claims, these electrodes should have about 10**17 He atoms. This would be an appropriate section in which to deal with Schwinger's hypothesis - cold fusion is real, but it comes from the p + d reaction. But where is the 3He?

- 3. We should supplement the gamma spectrum published by Fleischmann & Pons by an authentic gamma spectrum from neutron capture by protons to give deuterons plus gamma. One can use either the Petrasso spectrum in this region or a spectrum given us by Lewis and Barnes at Cal Tech. An important point here not mentioned in Schiffer's draft is the Fleischmann-Pons spectrum does not have a low energy Compton satellite. I am sending Schiffer a copy of the two spectra. I am sure that the Graphic Arts Division at Argonne can reproduce these to the same scale and then superimpose them.
- 4. Change reference In from Bombay to Bhabha.
- 5. Section Vc. The reference is to Scott at ORNL. He does not describe the closed cell explicitly in his memo to Appleton, but it is not ruled out. I actually learned the details of his and all the ORNL work on my visit to ORNL on 12 September. I have sent a separate memo to Alan Bard giving him a summary of the calorimetry. There are no smoking guns.
- 6. Section Vd. Second paragraph. is lower by 0.6 than the D2O (liquid).
- 7. Section Vd. The tritium production rate in the Bockris cells (Bockris, I don't know about Kevin Wolf) is $> 7 \times 10 \% 9/\text{sec}$. Thus the secondary neutron yield is > 10 % 5/sec. Change 10,000 to 100,000 unless you have a lower tritium production rate from Kevin Wolf in the actual cells in which he measured the 1 neutron/sec.
- 8. Section Vd. Regarding Martin's work. Johnson & Mathey, a major supplier.
- 9. Section Vd. Change the last sentence in the last paragraph to read: "Additional investigations are desirable....observed."
- 10. Tables I and II. The entrance in the header $2 \times 10*** 11$ neutrons (protons) per DD pair per sec. required by 1 watt of heat production is not clear.
- 11. The Walling Simons paper has been published in the Journal of Physical Chemistry.

Jacob Bigeleisen

BITNET copies to: Garwin, Hoffman, Huizenga, Koonin and Schiffer -----

Date: Tue, 26 Sep 89 13:57 EDT From: JBIGELEISEN@SBCCMAIL.BITNET Subject: Additional Comments

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Jacob Bigeleisen Professor Chemistry 516-632-7905 26-Sep-1989 01:49pm EDT

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SUBJECT: Additional Comments

John:

I believe you need to expand on the Sandia results in Section IIId. This is just not a question of not reproducing the results; the Sandia experiment shows what the problem is with Menlove's bursts. Menlove has not been too receptive to critical analyses of his neutron detection. He was cool and unreceptive to a suggestion from Maurice Goldhaber to replace the 3He with 4He in half of his counters. He said the counters are sealed commercial units. It takes too long to order 4He filled counters. With the great LANL shops he could have buit some 4He counters. By this time he should have been able to have received ones from a commercial vendor.

There is no entry in Table I for the Sandia experiments. Reference [16] in Appendix IIIc should be changed to [Bi]. JAKE

9 N

Energy Research Advisory Board

to the United States Department of Energy 1000 Independence Avenue, S.W. Washington, D.C. 20585 (202) 586-5444



September 25, 1989

Subject:

Chairman's July 20 Request for Summaries of Past and Present Cold

Fusion Research and August 9 Request for Tritium Production

Results

The following have been received to date from the following individuals in response to the above requests:

S. Jones, et.al, Brigham Young University (response to August 9 request) D.R. McCracken, AECL (response to August 9 request) XYZ.

BEGIN RED SERIES

- J.F. Holzrichter, LLNL (response to August 9 request) Α.
- W. Appleton, ORNL (response to July 20 request) В. D. Manos, PPPL (response to August 9 request)
- С. K.I. Thomassen, LLNL (response to July 20 request) D.
- M. Danos, NIST (response to July 20 request) Ε. R. Jensen, LANL (response to July 20 request) F.
- J. Harb & W. Pitt, Brigham Young U. (response to August 9 request) G.
- A. Narath, Sandia National Laboratories (response to August 9 request) Η.
- S. Pons, U. Utah (response to August 9 request) Ι.
- H. Randolph, Westinghouse Savannah River Co. (responses to July 20 and J. August 9 request)
- N. Samios, Brookhaven National Laboratory (response to July 20 request) Κ.
- C. White, Naval Research Laboratory (response to July 20 request) L.
- H. Law, AT&T Bell Laboratories (response to August 9 request) Μ.

Immediately behind this memorandum is a cumulative index.

. Woodard Panel Secretary

Enclosures

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INDEX

- A. Bo U.R. Sundqvist, Uppsala University (response to both requests)
- B. W. Scheid, Giessen University (responses to both requests)
- C. M.M. Broer, AT&T Bell Laboratories (response to August 9 request)
- D. J. Eridon, Naval Research Laboratory (response to August 9 request)
- E. Y.E. Kim, Purdue University (response to July 20 request)
- F. A Schriesheim, Argonne National Laboratory (response to July 20 request)
- G. M.A. Prelas, University of Missouri, Columbia
- H. L.R. Greenwood, ANL (response to August 9 request)
- I. F. Besenbacher, University of Aarbus (response to July 20 request)
- J. N.J.C. Packham (for Bockris), Texas A&M (response to August 9 request)
- K. J. Paquette, Atomic Energy of Canada, Ltd.
- L. N. Lewis, California Institute of Technology (response to August 9 request)
- M. J. Rafelski, University of Arizona (response to August 9 request)
- N. P.D. Bond, Brookhaven National Laboratory (response to August 9 request)
- O. J.L. Straalsund, Battelle (response to August 9 request)
- P. A. Narath, Sandia National Laboratories (response to August 9 request)
- Q. W. Appleton, Oak Ridge National Laboratory (response to August 9 request)
- R. Texas A&M University (response to August 9 request)
- S. T.F. Droege (response to July 20 request)
- T. S. Lien, Idaho Operations Office (response to August 9 request)
- U. M. Wadsworth, U. Utah (response to August 9 request)
- V. J. Farny, Institute of Plasma Physics, Warszaw (response to July 20 request)
- W. S. Jones, et.al, Brigham Young University (response to August 9 request)
- XYZ. D.R. McCracken, AECL (response to August 9 request)

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- A. J.F. Holzrichter, LLNL (response to August 9 request)
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